TEXTILE RESEARCH JOURNAL

VOLUME XXIV | May, 1954 | NUMBER 5

Annual Review Issue

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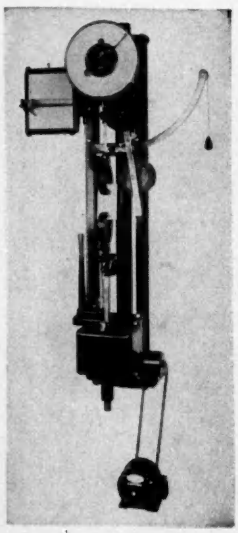

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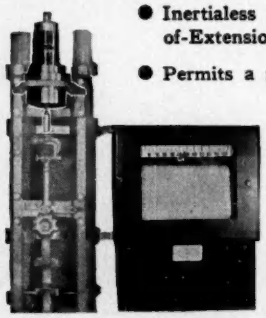

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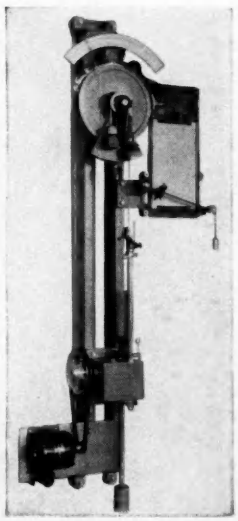

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In This Issue

A New Method for Differentiation of Skin and Core of a Rayon Filament
W. Rhett Berry 397

Degradation of Cotton Fibers and Yarns by Heat and Moisture
Rollin S. Orr, Louis C. Weiss, Geraldine C. Humphreys, Trinidad Mares, and James N. Grant 399

An Improved Strain Gage for Precise Force Measurement in Single Fibers
Walter J. Thorsen 407

The Detection of Modification in Animal Fibers
Harris M. Burte
II. Tensile Recovery at 65% R.H. 414
III. Tensile Recovery in Water 423

Letters to the Editor
Static Electrification of Filaments 426

INDUSTRIAL SECTION

Efiect of Single and Ply Twists on the Properties of a 15.5/2 Carded Cotton Yarn
Louis A. Fiori, John J. Brown, and Jack E. Sands 428

The Effect of Cap-Edge Friction on Spinning
J. Crank and Doreen D. Whitmore 434

Review of Textile Research and Development During 1953
Edited by Richard Steele, Corneil Messler, and R. K. Toner 439

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TEXTILE RESEARCH JOURNAL, published monthly at Prince and Lemon Streets, Lancaster, Pa., is devoted to the interest of research in textile and allied industries. It is published by Textile Research Institute.

Entered as second-class matter, April 27, 1945, at the post office at Lancaster, Pa., under the Act of August 24, 1912. Accepted for mailing at special rate of postage provided for in paragraph (d-2), Section 34.40, P. L. & R. of 1948, authorized May 9, 1950.

Manuscripts and communications should be sent to the editorial office, 10 East 40th Street, New York 16, N. Y.

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TEXTILE RESEARCH JOURNAL

VOL. XXIV, NO. 5 MAY, 1954

A New Method for Differentiation of Skin and Core of a Rayon Filament

W. Rhett Berry

The American Enka Corporation, Enka, North Carolina

Abstract

A new and rapid technique for staining the skin of a rayon filament and hence for determining the skin area is described. The technique requires the combined use of two dyes, Solophenyl Fast Blue Green BL. (Geigy) and Aniline Gentian Violet.

It is known that the Solophenyl Blue Green dye will only stain the core of a viscose filament. Upon the addition of Gentian Violet, the blue-green dye will be removed from the core and the skin will be stained a violet color. For the test to be successful, it is necessary that the core of the filament be stained first with the blue-green dye before the violet is added to stain the skin.

This technique is found to be less time consuming than the Victoria Blue technique for selective skin dyeing as described by Morehead and Sisson [2] or the selective staining of the skin described by P. H. Hermans [1].

It is probable that only physical absorption of the dyes by the skin and core is involved.

Introduction

Previously, a number of techniques for the selective staining of skin and core of a viscose filament have been described. However, in each case, the technique was fairly time consuming, ranging from 3 hrs. to overnight staining, or required the use of expensive solvents in an involved procedure. Thus, the desirability of having a quick, easy method for staining the skin of rayon cross sections led to the development of the staining method described herein. It requires from 5 to 10 min. for the complete staining process and the use of water and denatured alcohol as solvents.

This method has the virtue that apparently no chemical action takes place between the dyes and cellulose but only the physical condition of the cellulose determines the dye retention in the skin.

Experimental Procedure

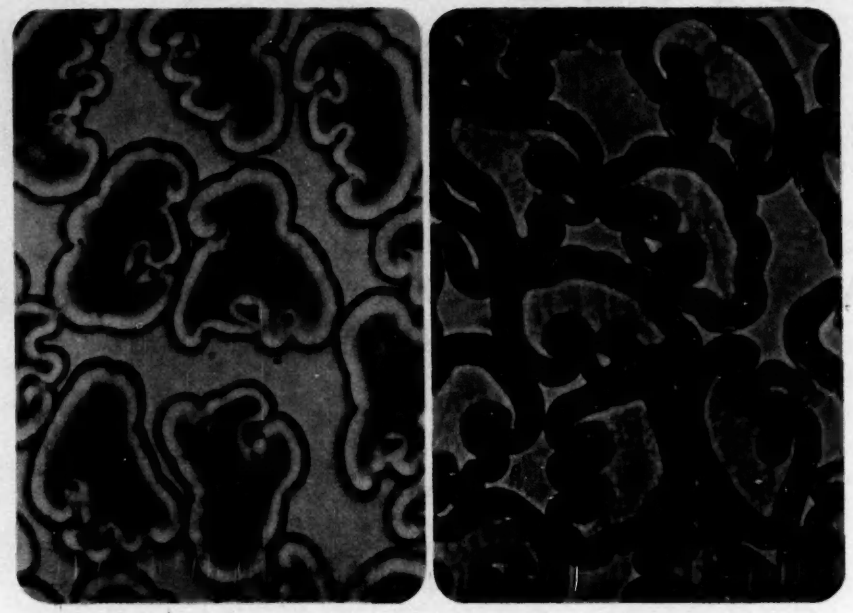
In this staining method, paraffin embedded cross sections of 5μ to 6μ thickness were used. The sections were fixed on the slide by the usual adhesive of egg white, glycerine, and water. Xylene was used as a solvent for the paraffin.

The steps in the staining technique are as follows:

(*1*) First flood the slide with 10% aqueous solution of Solophenyl Fast Blue Green BL. (Geigy) and steep for 30 to 45 seconds over a low flame. (This dye is water soluble and alcohol insoluble.)

(*2*) Wash excess dye from slide by dipping in a beaker of water.

(*3*) Flood the slide with Aniline Gentian Violet and steep for 30 to 45 seconds over a low flame. (This dye is alcohol soluble and water insoluble.) The solution is made as follows: Saturated solution of Gentian Violet in denatured alcohol (95% ethyl, 5% methyl)—24 cc.; denatured alcohol—20 cc.; aniline water—200 cc. It was found that the 94% Crystal (Gentian) Violet was most desirable to use in this procedure. The aniline water is prepared by shaking 4 cc. of aniline with 196 cc. of water for several minutes. It should then be filtered through



a b

FIG. 1

filter paper until clear; this results in a practically saturated solution. The Gentian Violet solution will last from 2 to 3 weeks.

(*4*) Wash the slide in a beaker of denatured alcohol for 5 to 10 seconds (or longer if necessary) examining to see when the core washed free of dye.

(*5*) Wash in water to stop alcoholic washing action.

If the slide is examined after step no. 2, it will be seen that the cores of the filaments have been stained blue while the skins remain unstained. This is shown in Figure 1a.

Examination after step no. 3, but before the alcohol washing, step no. 4, reveals that the core is more deeply stained than the skin, the core being almost black and the skin a deep purple. In step no. 4, the washing time is very critical and the slide should be examined in order to stop the alcoholic washing as soon as the cores have been washed free of dyes. Normally, it requires only 5 to 10 seconds (longer in some cases) to wash the core, leaving the skin stained a brilliant purple. This is shown in Figure 1b.

The slide is then ready for mounting and examination. It should be mentioned that in all washing steps, the slide should be very gently agitated so that as few of the sections as possible will be lost.

If the alcoholic washing in step no. 4 is continued too long, examination of the slide reveals that the Gentian Violet in the skin washes out and leaves the filament completely unstained. This action indicates that there is no chemical reaction taking place between the dye and the cellulose but rather a physical absorption of the two dyes by the skin and core of the filaments.

Discussion

The question as to the necessity of the use of both dyes is interesting. Why not begin with step no. 3 by flooding the slide with the Gentian Violet, proceed with the alcohol wash in step no. 4, and have the skin stained? Experiment shows that the staining method will not work without first adding the Solophenyl Blue Green (step no. 1). If only the Gentian Violet is added, the entire filament stains (skin and core). On washing in alcohol the dye washes out evenly from skin and core, leaving the filament unstained.

A possible explanation of this peculiar phenomenon is as follows: the core of the filament has a greater

affinity for the Solophenyl Blue Green dye than does the skin, perhaps due to the difference in structure of the skin and core. When the blue dye is added, the filament core absorbs the dye until it reaches its saturation point. On adding the Aniline Gentian Violet, the unstained skin of the filament absorbs the violet dye, and a reaction between the blue dye in the core and the violet takes place, forming a crystalline precipitate of unknown composition. The answer to the exact nature of the above reaction may lead to additional information concerning skin and core structure. Examination at this point reveals the core to be darker than the skin. On washing in alcohol, the precipitate washes out, leaving the skin stained purple. The phenomenon designated by P. H. Hermans as "anisotropic diffusion" may also be a partial explanation [1].

After the skin has been stained, it is possible to revert to the condition shown in Figure 1a by flooding the slide after step 5 with Blue Green Geigy and heating. On examination after washing in water, it will be found that the core has been stained as in step no. 1, and the Gentian Violet has been removed from the skin.

The contrast of the purple stained skin is sufficiently great to make black on white photographs without the use of special filters [1, 2].

Acknowledgment

This work was done in the Enka Plant Microscopical Laboratory under the direction of Mr. E. C. Jolliff, whose continued interest in this development the author wishes to acknowledge.

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1. Hermans, P. H., TEXTILE RESEARCH JOURNAL **18**, 9 (1948).
2. Morehead, F. F., and Sisson, W. A., TEXTILE RESEARCH JOURNAL **15**, 443 (1945).

(Manuscript received October 29, 1953.)

Degradation of Cotton Fibers and Yarns by Heat and Moisture*

Rollin S. Orr, Louis C. Weiss, Geraldine C. Humphreys, Trinidad Mares, and James N. Grant

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Abstract

Strength and elongation measurements were made on single cotton fibers and on yarns which had been subjected to various temperatures from 110° to 162°C and various moisture conditions from 3% R.H. up to saturation for periods of heating from 2 to 128 hrs. Moisture contents and degrees of polymerization were also determined, the latter being used to calculate cellulose chain rupture.

The simultaneous reduction in strength and elongation at break indicates that heat degradation weakens fibers by creating or intensifying weak points along the fiber. An equation similar to that derived by Sippel, relating fiber strength loss to time of heating and percentage of cellulose links broken, is discussed. Yarn strength, although not as readily affected by heat degradation as fiber strength, follows a similar pattern.

Introduction

An understanding of the process by which cotton is degraded by heating in the presence of air and moisture is important in finishing processes, in laundering, and in connection with certain end-uses where the service is at elevated temperatures, such as conveyor belts and ironing-board covers.

As shown by others, heating produces permanent losses in breaking strength, elongation at break, and degree of polymerization of both cotton and rayon

* Presented in part before The Fiber Society, Fontana, N. C., Apr. 27, 1950.

† One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

yarns and cords [4, 5, 10, 13, 26, 27, 29]. It reduces the ability of cellulose to absorb moisture and dye [12], increases the density [20], and changes the pH and electrical conductivity [14].

The rate at which heat degrades cellulose is not appreciable at 100°C or less, but increases rapidly as the temperature is increased [27, 29]. The presence of moisture during heating has been found to accelerate degradation [9, 10, 16, 26, 29], while the exclusion of oxygen by evacuation [10, 26, 29] or by extremely high humidities [9] seems to reduce the rate of degradation. Protective coatings, such as natural waxes [26, 29] and rubber coatings [4], have been found to reduce degradation.

The strength of heat-degraded tire cords is well correlated with the percentage of links broken in the cellulose molecule as estimated from the degree of polymerization [4] based on viscosity measurements. Complete loss of tire cord strength was found [4] when only about 0.3% of the links were broken in cotton cord and about 0.6% in viscose rayon cord.

Only minor changes in stress-strain modulus [24] and in elongation at 10-lb. load [4] of heat-degraded cotton tire cords have been noted, indicating a concurrent reduction in strength and elongation at break.

While these previous studies have revealed the influence of heat degradation and moisture on many properties of cellulose and on the physical properties of cords and yarns, their influence on the strength and elongation of the individual fibers and the interrelationship of these properties to those of yarns have not been studied.

In order to obtain a better understanding of the fundamentals involved, the present studies were made both on single fibers and on yarns. Due to fiber slippage and the effects of twist, the strength and elongation values for yarns and cords differ from those of their component fibers. Therefore, more direct information on degradation may be expected from studies of single fibers than of yarns and cords.

The present investigation covered certain interrelations between strength, elongation, calculated percentage of links broken, secant modulus, moisture content after heating, time of heating, degrading temperature, and moisture content during heating for fibers and yarns.

Experimental Methods

Sampling

A well-blended sample of Stoneville 2B cotton was used, from which were taken the unprocessed fibers and cotton for processing into 16/2s yarn. For studies of single fibers, the unprocessed fibers were arrayed into groups according to length using the A.S.T.M. method [1]. Fibers of the three adjacent length groups containing the largest total weight of fibers were well blended to produce a sample of approximately 260 mg. The blended sample was divided into 128 subsamples through successive division of the sample. The final subsamples of about 2 mg. each were gently combed and fastened between two sheets of thin porous paper. Although the paper probably increased the time required to reach atmospheric equilibrium conditions, it prevented contamination of the fibers by any abrasive grit which could cause physical damage.

The 16/2s yarns with Z/S balanced twist were taken from the spool as follows: approximately 800 yds. of yarn were wound in a 54-in. skein; this was then cut into three lengths, producing 1,600 specimens of an 18-in. length. For the tensile tests, these yarns were divided by random selection into 80 samples of 20 specimens each. Each group of 20 yarns was loosely coiled and fastened between sheets of thin porous paper. No extra precaution was taken to prevent loss of twist, since, due to the balanced construction, such loss was considered negligible. Furthermore, any changes would be common to both heated yarn and the control.

For viscosity determinations, samples of the yarn were prepared by a similar procedure.

Apparatus

The forced draft oven and the air humidifier unit used in this study were similar to those previously described [7]. The oven was controlled within a precision of ± 2°C, using two independent thermoregulators to control electrical heating elements.

The dew point of the air from the humidifier was controlled within a precision of ± 1°C. The humidified air was brought to oven temperature while passing through 7 ft. of ⅜-in. copper tubing within the oven and then through a glass container in which the samples were arranged. The exit of the container was plugged with loose cotton to prevent any decomposition products in the oven air from re-entering the container. Since the flow of air was about 1 c.f.m., a complete change of air was effected every few seconds. This rapid exchange of air is believed to have reduced the induction period required to establish the constant temperature and moisture conditions around the sample.

Treatments

The fibers and yarns were heated at oven temperatures of 110°, 138°, and 162°C in air having at each temperature dew points of 25°, 55°, 80°, and 100°C. These represent relative humidities of 3.1%, 15.5%, 46.8%, and 100%, respectively, as calculated from $100p/P$, where p is the vapor pressure at the dew points and P is the pressure of steam at 100°C—*i.e.*, 760 mm. of Hg. This is in conformity with the concept of Dillon and Prettyman [6] for relative humidities at temperatures above 100°C and atmospheric pressure. The 100°C dew point was obtained by passing steam into the oven. The periods of heating were 4, 8, 16, 32, 64, and 128 hrs., except that for the oven temperatures of 162°C a 2-hr. period was substituted for the 128-hr. period. All samples were reconditioned for at least 2 weeks in a standard atmosphere before testing.

Tests

Breaking load and elongation at break were determined on 100 single fibers from each sample using the fiber tester and technique previously described [17]. The length of fiber subjected to tension was 7.1 mm. However, some measurements at 2.9 and 0.9 mm. were also made on unheated fibers and on fibers degraded to about 50% of the original strength. Load-elongation curves were made on 12 additional single fibers from each degraded sample with a single-fiber tester of a similar design [18].

Load-elongation curves were made on 20 yarn specimens from each sample, using a standard constant-rate-of-loading tester of 2-kg. capacity.

From cuprammonium fluidity measurements made on selected samples, the percentage of links broken was computed for the degraded yarns according to the method used by Conrad, Tripp, and Mares [4].

Moisture contents after degradation were determined by the standard A.S.T.M. method [2] on the 1.5-g. samples of cleaned and mixed unprocessed cotton which had been heat-degraded and allowed to recondition for 2 weeks at standard conditions. Moisture contents during degradation were measured on 2-g. samples of yarn which were exposed to the degrading condition for 2 hrs.

The length and weight of yarns were determined before and after heating at 162°C for periods up to 24 hrs. at 3.1% R.H. The length was measured under a load equal to 10% of the breaking load. The yarns were allowed to recondition at standard conditions for 1 week; they were then reweighed and the moisture content was determined.

TABLE I. PHYSICAL CHARACTERISTICS OF THE UNHEATED COTTON FIBERS AND YARNS

Physical property	Fibers	Yarns
Breaking load (g.)	4.8	1570
Weight fineness (grex)	1.9	910
Tenacity (g./grex)	2.5	1.7
Elongation at break (%)	6.7	7.4
Secant modulus* ((g./grex)/strain)	37.0	22.0
Degree of polymerization†	4990	
Moisture content (%)	6.7	

* Average slope of the stress-strain curve between 0.2 g. and load of rupture for fibers, and between 200 and 600 g. for yarns.

† This, in conjunction with similar data for degraded celluloses, was used as the basis for calculating percentage of links broken according to Conrad *et al.* [4].

Results

In Table I are given the values for the physical properties of the unheated fibers and yarns. In calculations of tenacity, differences in weight fineness of unheated and heated yarns and fibers were neglected. The decrease in length was less than 0.4% and the decrease in weight was only about 1.5%, 0.5% of which was due to loss in moisture.

In the graphs presented here, the values for the degraded cotton are usually plotted as percentages of the corresponding values for the unheated fibers or yarns.

Strength-Elongation-Time Relations

In Figure 1 are given the semilogarithmic relations of retained strength of single fibers to time of heating for the different oven conditions; in Figure 2 similar relations for yarns are given. The curves in both of these figures show that the rate of loss of strength decreases with period of heating. At the higher temperatures increasing moisture increases the rate of degradation, except at 100% R.H., where the samples were degraded in an atmosphere of steam which was essentially oxygen-free. The moisture contents of the yarns during degradation at the various temperatures and humidities are given in Table II.

The relations of elongation at break of fibers to periods of heating were similar to the relations of retained strength of fibers to periods of heating. This suggests a close relation between strength and elongation at break. In Figure 3 the average breaking load of fibers at each condition is plotted as a

TABLE II. MOISTURE CONTENT OF COTTON YARNS DURING DEGRADATION *

Temperature (°C)	Moisture content (%) at: 3% R.H.	16% R.H.	47% R.H.	100% R.H.
110	0.10	1.20	2.40	3.50
138	0.00	0.35	1.00	1.85
162	0.00	0.10	0.20	0.60

* Moisture content as determined by A.S.T.M. oven-dry method, which would not remove bound water.

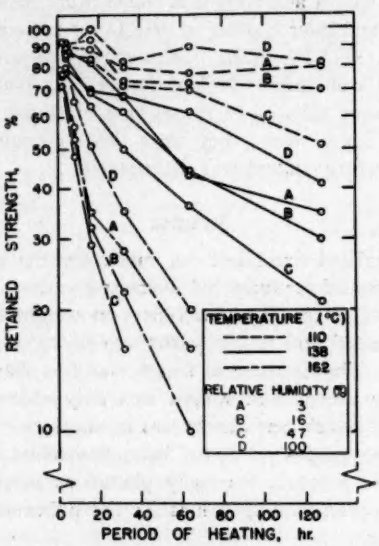

FIG. 1. *Relation of retained strength of single cotton fibers to period of heating at various combinations of temperature and moisture.*

function of the corresponding elongation at break. A simultaneous decrease in strength and elongation at break is indicated. Average load-elongation curves of unheated fibers, *A*, and fibers degraded to about 35% retained strength, *B*, are included for comparison. The average load-elongation curve of unheated fibers is essentially the line of best fit for the points of average retained strength and elongation.

Since the weight fineness was constant, this concurrent reduction in strength and elongation at break indicated that no change in the secant modulus of the fibers took place with degradation. Since the load-elongation curves of the single fibers are nearly linear (Figure 3), the ratio of breaking stress to breaking strain was taken to be the average secant modulus.

In the calculation of secant modulus for yarns, the effect of kinkiness of the fibers and the opening up

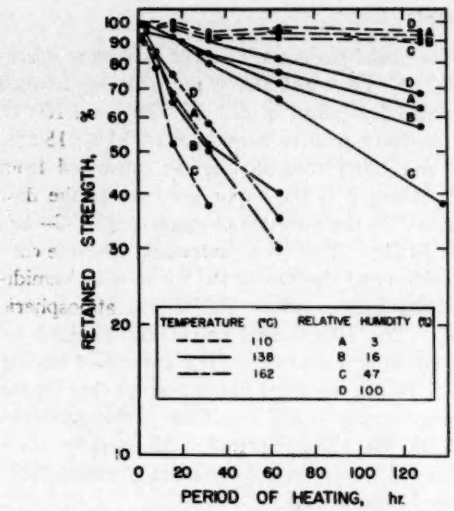

FIG. 2. *Relation of retained strength of cotton yarns to period of heating at various combinations of temperature and moisture.*

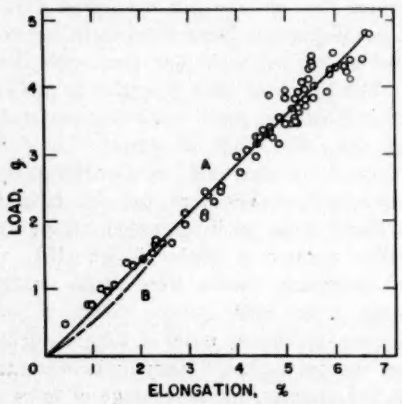

FIG. 3. *Breaking load and elongation at break for heat-degraded cotton fibers, with superimposed load-elongation curves for unheated fibers,* A, *and highly degraded fibers,* B.

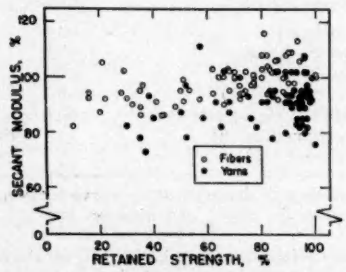

FIG. 4. *Relation of secant modulus to retained strength of degraded cotton fibers and yarns.*

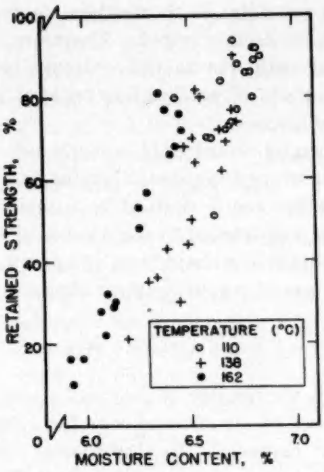

FIG. 5. *Relation of retained strength of fibers to moisture content of heat-degraded cotton.*

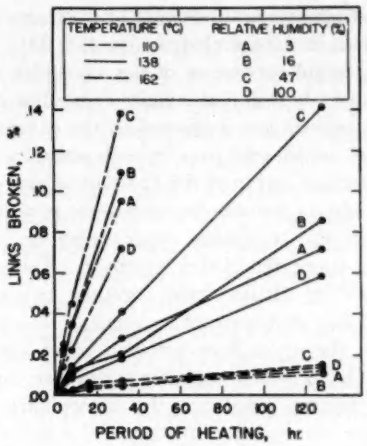

FIG. 6. *Relation of cellulose links broken to periods of heating at various combinations of temperature and moisture.*

of the yarns when heated are reduced by disregarding the lower part of the load-elongation curves. They were calculated between loads corresponding to 12% and 36% of the strength of the unheated yarn. The secant modulus of both fibers and yarns is shown (Figure 4) to be nearly independent of loss in strength. The slight reduction in secant modulus with decreasing strength is not explained by the lower moisture content of the degraded samples, since decreased moisture generally increased modulus.

From Figure 5 it is observed that moisture content of samples at the same retained strength is generally lower, the higher the temperature during heating.

Percentage of Links Broken

In Figure 6 the relation of percentage of links broken is given as a function of period of heating for various conditions for degradation. The nearly linear relation illustrates that after an induction period of several hours, the rate at which links are broken is nearly constant for each condition. If the rate of link breakage is directly proportional to the number of available links, this nearly linear relation will be expected to hold only so long as the percentage of available links broken is small. A fraction of 1% of links broken has been reported as sufficient to cause complete loss of strength [4, 22]. The departure from linearity and the higher initial rate of link breakage for short periods of heating may have been due partly to the initial moisture content of about 5%, which is greater than any during heating.

Discussion

The concurrent reduction in strength and elongation at break of fibers suggests a possible mode of action in degradation. If a long rod is weakened by making shallow cuts perpendicular to its axis, the load-elongation curve which can be obtained on stressing will be altered only at its upper end and the slope at points along the load-elongation curve will remain nearly constant. This is in contrast to the effect of slicing or cutting the rod parallel to its axis. The proportional reduction in strength and elongation at break of fibers (Figure 3) and the constant secant modulus of fibers and yarns regardless of retained strength (Figure 4) indicate that the fibers may be weakened in the first manner—*i.e.*, by the development of cracks or weak regions of short depth along the length of the fiber.

Photomicrographs of the cotton fibers degraded to less than 30% of their original strength and swollen in 18% NaOH clearly show (Figure 7) transverse cracks in the cellulose. This has also been reported by Bailey [3]. Similar transverse cracks have been reported in hydrolyzed ramie fibers [8].

The effect of such weak points as a factor in determining the strength even of undegraded cotton fibers is shown by the unusually large coefficient of variation in strength and elongation at break, and by the large decrease of strength with increase of test length [19]. The presence of weak points in undegraded cotton may be due to mechanical damage in process-

ing, to strains set up in drying, or to strains at points of reversal in the spiral structure [15, 25].

The general conception of the molecular structure of cellulose fibers is that definite crystalline or highly ordered regions are connected by the cellulose chain molecules, which may pass through several such crystalline regions [4]. If the crystallites and the connecting chains are aligned somewhat parallel to the fiber axis, the crystalline regions may be compared to knots tying the chains together. This network of connecting chains, held together by crystalline "knots," may then account for much of fiber strength. Between the crystallites are also other chains and ends of chains which, together with those connecting the crystallites, constitute the amorphous regions, which are more subject to chemical action than the crystallites. According to this conception, strength reduction by degradation is due in large part to rupture of the chains connecting the crystallites. Although these connecting chains are in a direction generally aligned with the fiber axis, their length between crystallites is short when compared to the length of the section tested. Therefore, the reduction in strength due to their rupture is consistent with the effects of weak points on fiber strength as previously discussed.

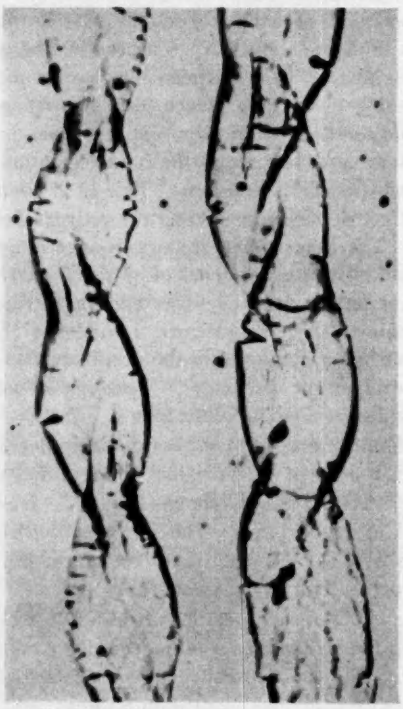

FIG. 7. *Photomicrograph showing transverse cracks in highly degraded cotton fibers in 18% NaOH. (Magnification, 500 ×.)*

As shown by Sippel [21], a mathematical relation of the fiber strength to time of heating and percentage of links broken can be derived by assuming that fiber strength is proportional to the number of unruptured connecting cellulose chains in a cross section of fiber. The time rate of rupturing these chains is then

$$\frac{dH}{dt} = r(N - H), \tag{1}$$

where H is the number of ruptured chains, N is the total number of chains, and r is the rate at which chains are ruptured. By considering the rate, r, constant for a given temperature and humidity, as shown in Figure 6, integrating between the limits of $t = 0$, t and $H = H_o$, H, equation (1) would then give

$$\ln\left(\frac{N - H_o}{N - H}\right) = rt, \tag{2}$$

where t is the time heated, and H_o is the number of ruptured connecting chains at the beginning of the degradation treatment. From the assumption that strength is proportional to the number of unruptured connecting chains, it follows that

$$\ln\left(\frac{S_o}{S}\right) = rt, \tag{3}$$

where S_o is the original fiber strength and S is the degraded strength. This may be expressed in the form used to plot Figures 1 and 2:

$$\log\left(\frac{100S}{S_o}\right) = \log 100 - rt. \tag{4}$$

As shown by Figure 6, the percentage of links broken, K, is proportional to rt, so that

$$\log\left(\frac{100S}{S_o}\right) = \log 100 - bK, \tag{5}$$

where b is a constant.

The data for links broken and retained strength were plotted according to equation (5) in Figure 8. The fact that the single fiber curve does not pass through 100% retained strength at zero percentage of links broken may be due to the rapid initial rate at which links are broken during the induction period.

Others have reported that all sensible tenacity is lost at a degree of polymerization between 300 and

100 [4, 22], which would correspond to the breakage of between 0.3% and 1% of the links in cotton. The relations shown in equations (4) and (5) would indicate that when 1% of the links are broken, the single fiber strength would be less than 0.001% of the original strength, and yarn strength would be less than 0.01% of the original strength.

The results obtained indicate the relation of fiber strength to yarn strength. While strength was determined on the fibers from three modal length groups in a sample, these groups comprise 40% to 65% of the fibers in the sample and are those considered to be largely responsible for yarn strength. The length of fiber section tested is not greatly different from that commonly believed to be the effective length in these yarns. Furthermore, for the degraded fibers the effect on retained strength of length of fiber section tested is small, as shown by the tests made at 0.9, 2.9, and 7.1 mm.; retained strengths were 53%, 45%, and 47%, respectively.

Any differences which existed in the form or preparation of fibers and yarns for heating were not expected to cause a difference in the rate of degradation. Other than a possible small change in coefficient of friction caused by heating, the results are believed to represent the relation of fiber strength to yarn strength after a degradative reaction.

In theoretical studies, the assumption has been made that yarn strength is a linear function of fiber strength [11, 23]. However, the statistical studies of Webb and Richardson [28] have shown for untreated cotton measured by the Pressley bundle method that *beta* correlation coefficients of yarn strength with fiber strength are only about 0.36. The plot of fiber strength *vs.* yarn strength for heat-degraded cotton in Figure 9 indicates that the relation is curvilinear and that a 30% decrease in fiber strength causes only a 10% decrease in yarn strength, while a 50% decrease in the fiber strength causes a 20% decrease in the yarn strength.

Since fiber strength and elongation can be varied by heat degradation with little effect on other properties of the fiber, further studies of fiber-to-yarn relations in which all properties except tenacity and elongation are held constant can be made.

The prominent effect of moisture in heat degradation should be a consideration in laundering and other high-temperature operations where moisture is present. For least degradation the moisture should be very low or high enough to exclude oxygen—*i.e.*, near 100% relative humidity.

Conclusions

It has been shown that increases in both temperature and moisture are associated with increases in the rate of degradation of both fibers and yarns, except that exclusion of oxygen by relative humidities near 100% reduces rate of degradation. The time rate of loss of remaining strength is approximately constant.

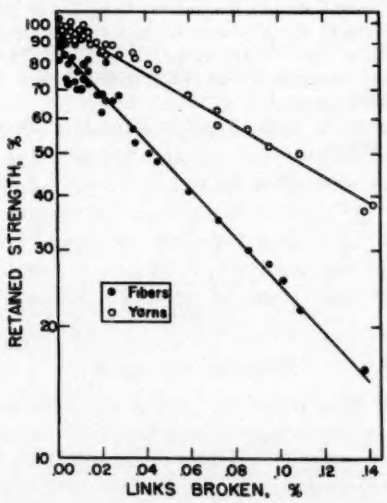

FIG. 8. *Relation of retained strength of single cotton fibers and yarns to cellulose links broken by heat degradation.*

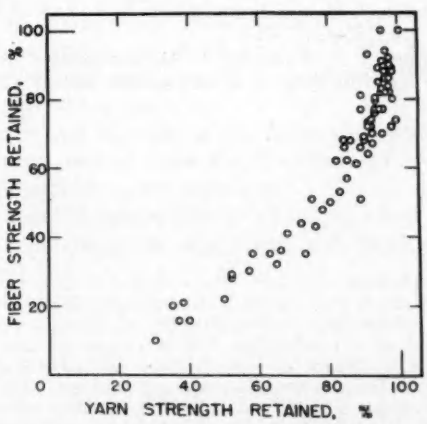

FIG. 9. *Relation of fiber strength retained to yarn strength retained for heat-degraded cotton.*

The simultaneous reduction in strength and elongation at break indicates that heat degradation weakens the fibers and yarns by creating additional weak points along the fiber length.

The data indicate that yarn strength is not as readily affected by heat degradation as is fiber strength.

The interrelations of strength, elongation, time, and percentage of links broken are in agreement with the accepted concept of fiber structure.

Acknowledgments

The authors wish to express their appreciation to Cyril A. Caillet for moisture determinations; to New Products Section, Cotton Mechanical Processing, for supplying the yarns; and to Miss Ines V. deGruy for the photomicrograph.

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(*Manuscript received October 22, 1953.*)

An Improved Strain Gage for Precise Force Measurement in Single Fibers

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Abstract

An unbonded resistance wire strain gage is described that is sensitive to small forces, has high precision and accuracy, and is simple to construct. The low inertia and high stability of this gage make it especially suitable for the precise measurement of stress relaxation in single textile fibers over periods lasting from milliseconds to as long as 2 months or more. The output of the gage is sufficient for direct coupling to recording potentiometers.

Introduction

The precise measurement of the small forces in single textile fibers, filaments, yarns, and fabrics requires instruments that are sensitive, accurate, stable, and have low inertia characteristics. Forces can be measured in various ways [1, 2, 3, 5, 6, 7, 8, 9, 12, 13, 15] and resistance wire strain gages are employed frequently because of their simplicity in construction, accuracy, and precision. However, present resistance wire gages lack the high sensitivity, low inertia, and long-term stability required in stress-relaxation studies of single fibers; this is especially true in the case where measurements are desired which may range from a fraction of a second to long periods of time.

This paper describes the principle and construction of a resistance wire strain gage of sufficient sensitivity to operate directly recording potentiometers, with a precision greater than 3 mg., an accuracy and stability of better than ½% of full scale for periods longer than 5×10^6 seconds (2 months), and low inertia for measurements in the 10^{-3} second range. Force ranges from 0–1 to 0–20 g. full scale can be obtained, and its application to the measurement of stress relaxation in single wool fibers is shown.

Gage Development

Unbonded strain gages were constructed utilizing a variety of strain sensing configurations, each connected as a Wheatstone bridge circuit. All were tested for strain sensitivity, mounting stability, and stress hysteresis. Finally one configuration was chosen which is satisfactory in all these respects and yet simple to construct. However, tests of the gage showed that the resistance of the gage arms was not stable with constantan, stainless steel, or several commercial resistance wires. Therefore a search was initiated to locate a stable gage wire. The characteristics generally desired in such wires are: (*1*) high resistivity in order to (*a*) keep the current flow and consequent heat dissipation low, (*b*) make possible gages whose resistance is large compared to lead wires, and (*c*) allow small lengths of wire to be used in the gage, (*2*) low temperature coefficient of resistance (thus gage sensitivity to ambient temperature changes is low), (*3*) stable resistance, (*4*) high gage factor or unit change in resistance per unit change in length, (*5*) low thermal e.m.f. at copper terminals, (*6*) high creep strength and yield stress, (*7*) low hysteresis in stress cycles, and (*8*) availability of small sizes, in the order of 1 mil, so that appreciable elongations will be produced with the forces to be measured.

Piano wire has most of the above characteristics, but small enough sizes could not be obtained to compensate for its low resistivity.

A nickel-chromium alloy † [4] having a high yield stress and resistivity was tested and found stable

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† Nichrome V, a high grade, iron free, nickel-chromium alloy of 80:20 proportions is commonly used as the heating element material in high-temperature applications. It is also used for springs at high temperatures. The property which is primarily responsible for its stability in a strain gage is its high creep strength. The load required to produce a 1% elongation in 10,000 hrs. at 2000°F is approximately 200 p.s.i. Mention of proprietary names for equipment does not imply that the equipment is recommended as superior to others not mentioned.

over long periods of time under high stresses and above average gage current densities. Its application to and operation in the gage is described below.

Gage Construction

The gage consists of four unbonded elements of 1 mil (0.001 in.) Nichrome V resistance wire mounted as shown diagrammatically in Figure 1, and connected into a DC Wheatstone bridge circuit as shown in Figure 2. Wire *D* is the strain sensitive element. The force to be measured is applied at its center *E* through a thin glass rod *F*, approximately 1 mm. in diameter. The wires are approximately 20 cm. long. *D* is mounted with a 24° sag which gives a mechanical advantage of more than 2. When a force is applied to the glass rod, *D* elongates slightly. This increases its resistance and a small voltage output is produced. All connections "inside" the bridge proper must be carefully soldered because small resistance changes (see characteristics on pp. 411–12) cause unbalances or voltage output changes.

The four elements of the gage, *A*, *B*, *C*, and *D*, are silver soldered to brass tabs made from $\frac{1}{2}$-in. lengths of $\frac{1}{16}$-in. diameter brass tubing. The tabs are soft soldered to binding posts mounted on a micarta frame (see Figure 1). Thus broken or defective gage arms can be quickly replaced. Elements *A*, *B*, and *C* are mounted slack. The following special technique of soldering the Nichrome V wire to the brass tabs must be followed to ensure good and stable connections. A soldering mix is made of silver solder filings and silver solder flux proportioned about 3:1 by volume. The mix is dampened by adding a few drops of water. Then the wire is threaded through the tab and the tab mounted in a suitable holder. A small amount of the soldering mix is placed at the junction of the wire and the tab is heated gently with a small oxygen hand torch until the silver solder flows around the wire. In order to remove the flux crust, the arm can be immersed in 1*N* HCl for about 3 min., then rinsed in H_2O for about 4 hrs. These operations are greatly assisted by the use of a binocular microscope. A photomicrograph of a satisfactory soldered connection is shown in Figure 3. Since it is difficult to solder the elements to equal lengths, a short length of No. 36 (B. & S.) resistance wire * (usually about 2–6 in. in length) is added to the appropriate arm to bring the bridge to near balance. Resistors *M* and *N* (see Figure 4) are used for fine and coarse balance, respectively. *G* and *H* are single pole, silver contact, 11 position rotary switches,† *G* having $\frac{1}{4}$-in. lengths (0.0089Ω) of No. 24 Advance * resistance wire between its contacts and *H* having 2-in. lengths (0.071Ω) between its contacts. Resistors *O* and *P* are for exact zero voltage output

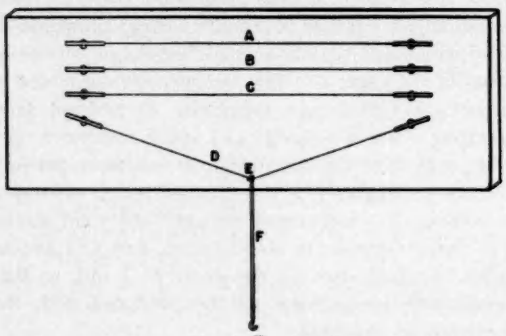

FIG. 1. *Diagrammatic sketch of strain gage.*

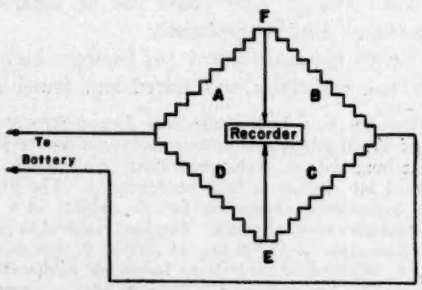

FIG. 2. *Basic gage circuit.*

* Advance or constantan may be used.
† The Mallory No. 1211L is satisfactory.

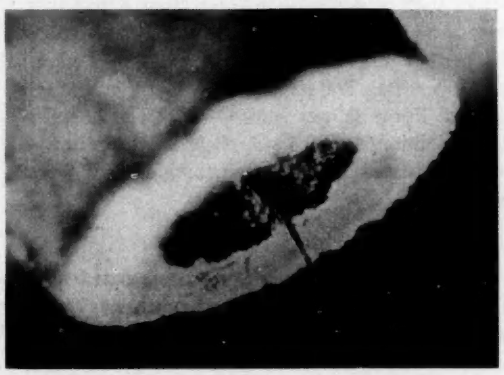

FIG. 3. *Photomicrograph of a satisfactory soldered connection between Nichrome V wire and brass tab. (Magnification 45×.)*

adjustment. O is No. 24 Advance resistance wire slightly greater than $\frac{1}{4}$ in. in length and P is a 1000Ω potentiometer. The resistance of P should be low enough to produce a gage output impedance considerably less than that of the recorder amplifier (see p. 411). R' is a 0–50Ω rheostat which is convenient for varying the gage sensitivity (mv. output/g.) to that of other gages, if more than one gage is used.

In addition to aiding the bridge balancing procedure, M and N also can serve to unbalance the bridge when desired in certain stress-relaxation experiments in which the forces to be recorded produce gage voltages larger than the full scale range of the potentiometer. For example, a 23 μ wool fiber will, on 20% elongation, exert an initial force of approximately 2.0 g. This force decays after a certain length of time to approximately 1.0 g. In order to observe this stress-decay most accurately, it is advantageous to operate the gages at the highest sensitivity possible. Since the sensitivity is directly proportional to the gage voltage input, it is best to operate the gage at as high an input voltage as possible. This limit was not determined, but voltages up to 24 volts were used without any difficulty. At 20-volts input the gage output is approximately 2.0 mv./g. and with the recording potentiometer set to record 2.5 mv. full scale it is seen that 2 g. of force will be off scale. However, resistors M and N can be tapped appropriately by switches G and H to unbalance the bridge or oppose this output voltage as desired to bring it within the range of the potentiometer. After the stress-relaxation experiment is over the fiber tension may be released and the gage calibrated with weights to determine the exact forces recorded after the "unbalancing" operation.

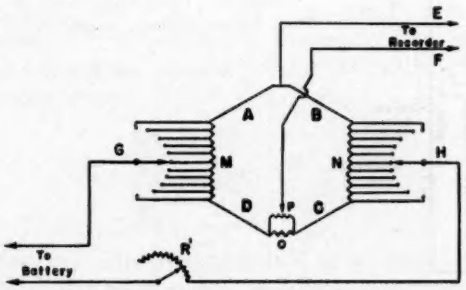

FIG. 4. *Complete operating circuit of the gage.*

Temperature Control

Since the temperature coefficient of resistance of Nichrome V is relatively high (A.S.T.M. values Oct. 1941, 0.000077 ohm cm. from 20°–250°C) it is necessary to thermostat the gage in order to obtain stability for longer than a few hours. This is best accomplished by mounting the gage within a thermostatted chamber. The temperature is controlled by copper tubes mounted on the inside of the chamber walls through which constant temperature water is circulated. All six walls are "covered" with coils because experience has shown that considerable air temperature drops occur near an uncovered wall, and the wall will vary in temperature as the room temperature changes. However, this wall temperature variation may be reduced by complete insulation outside, or if it is necessary to mount switches on one wall, insulation with glass wool on the inside of the wall or switch panel is sufficient. For precise control the water thermostat is mounted inside the gage chamber, itself mounted inside a container through which the water is circulated. Slight air currents will cause short transient gage output variations due to shifting of the layer of hot air around the wire. Therefore construction of the chamber is such that it is relatively draft-free, and a cover over the gage itself further reduces the possibility of drafts. Water temperature regulation to ± 0.02°C is sufficient.

Under the above conditions, after a 6-hr. warm up the gage has a precision and accuracy of ± 1.5 mg. at 24 volts input or better than $\frac{1}{2}$% of full scale for all force ranges and is stable (input voltage maintained constant) for periods of 2 months or more. The gage has been in use intermittently for over 2 years and no readjustment of the bridge balance resistor has been necessary due to zero shift. Due to its low inertia, forces have been measured as early as a few milliseconds after elongation. This modification will be discussed in a later paper.

Input Voltage Regulation

Without input voltage regulation, at 24-volts input (three 8-v., 80 amp.-hr. batteries) gage stability is satisfactory for only about 16 hrs. At lower voltages longer periods of stability may be attained. To extend the stability period another set of fresh batteries may be substituted and the "old" set recharged, etc., the exact input voltage being checked with a student's potentiometer and adjusted with a 0–5.0 ohm rheostat.

Special regulated 110 V A.C. rectifiers [14] have been used by others but through a modification of the principle used in recording potentiometers input regulation to better than 0.1% may be attained, which is very near the stability limitations of the standard cell. The method utilizes the basic potentiometric circuit shown in Figure 5. As the battery voltage drops, the unbalance detector together with a two-phase reversible servo motor [10] continuously decreases the resistance of the 0–5.0 ohm rheostat R_{bat}, thus keeping the voltage across the precision voltage divider R constant. Since the servo motor may drive the battery rheostat to its limit before the experiment is over, one should start with some resistance in the battery circuit which may be switched out as the voltage drops. It is preferable to do this automatically, and in order to reduce the drain on the standard cell, intermittent "balancing" and a "cut-out" for the standard cell is provided. This is accomplished as shown in Figure 6. When the rheostat arm has traveled nearly its full distance it activates a microswitch which switches the stepping relay to its next contact, thus decreasing the resistance in series with the battery by 4.0 ohms. The balance unit immediately brings the rheostat arm back to the same total resistance previously in the circuit, and the above sequence is slowly repeated as the battery voltage drops. The time switch actuates the balance unit every 30 min., connecting the standard cell at the same time, and the duration of each "balancing" operation is controlled by setting the time delay relay to any desired delay time, from 5 to 60 seconds. The ordinary relay is in its closed position during each cycle. When the timing switch turns the relays off it returns to its open position and the time delay relay returns to its closed position. Thus, neither the balance unit nor the standard cell is in operation except during the delay time of the time delay relay. The precision voltage divider is a 500 ohm precision multiturn potentiometer wound with Advance wire.

The constant balance unit or amplifier has an input "impedance" of 400 ohms and the standard cell has an internal resistance of less than 100 ohms. A 100,000 ohm resistor is switched in series with the

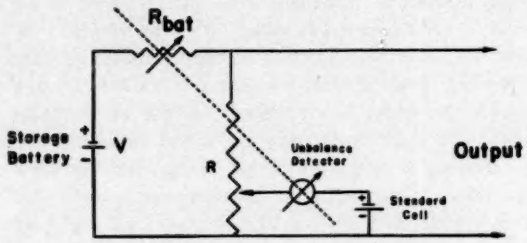

FIG. 5. *Basic potentiometric circuit of the automatic voltage regulator.*

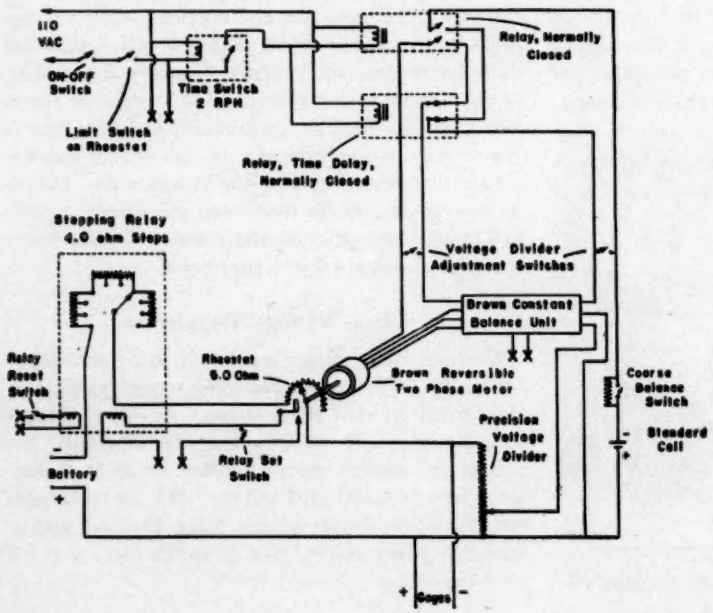

FIG. 6. *Complete circuit of battery voltage regulator.*

standard cell when making initial adjustments of the voltage divider in order to limit the current flow through the cell to less than 100 microamperes.

Recorders

Three main factors govern the selection of a recorder: (*1*) the pen speed desired, (*2*) the force range—*i.e.*, voltage range or span—to be measured, and (*3*) the output impedance of the gage.

Pen speed should be greater than the rate of change of force to be measured, and the recorder amplifier should have sufficient sensitivity to permit accurate recording at the span chosen.

The manufacturer of one recording potentiometer defines amplifier "impedance" as the resistance which if placed in series with a zero impedance voltage source across the input of the amplifier, would halve the sensitivity, or double the "dead zone" of the instrument. Therefore amplifier "impedance" should be high with respect to the gage output impedance.

A single point recording potentiometer with a 2-second (full chart traverse time) pen speed, a span of 2.5 mv., and an amplifier [11] with an impedance of 7000 ohms is used with the gage for stress-relaxation measurements in wool. Chart speeds are 30, 60, 90, and 120 in./hr. The amplifier has a sensitivity of 2×10^{-6} volts which allows full spans as small as 1 mv. and an accuracy of $\pm$ 0.2% of full scale.

Gage Characteristics

Bridge Equation

At the "balanced" condition (zero force, zero voltage output) the resistances A, B, C, and D (see Figure 2) are either equal or are related as follows:

$$\frac{A}{B} = \frac{D}{C}.$$

Assuming that A, B, C, and D are initially of equal resistance R, the relationship between the voltage output ΔE and the resistance change ΔR of the strain sensing arm is

$$\frac{\frac{\Delta R}{R}}{\frac{\Delta E}{E_B}} = 4 \quad \text{if} \quad \frac{\Delta R}{R} \ll 1,$$

where E_B is the voltage applied to the gage. This relationship holds as long as the input impedance of the recorder is very high with respect to the gage output impedance. This is true in the case of recording potentiometers which are null balance instruments; the actual impedance of these recorders at "balance" is essentially infinite.

Gage Sensitivity

Gage sensitivity is defined here as the voltage output ΔE per volt input per gram of force applied to the gage. Experimentally, at $E_B = 20$ volts, $\Delta E =$ 0.002 volt with a force F of 1 g. on the gage, or

$$\frac{\frac{\Delta E}{E_B}}{F} = 0.0001 \text{ g.}^{-1}.$$

Resistance Change of Strain Sensing Arm

From the above equations

$$\frac{\frac{\Delta R}{R}}{F} = 0.0004 \text{ g.}^{-1}.$$

Also, since $R = 430$ ohms, $\Delta R = 0.17 \frac{\text{ohms}}{\text{g.}}$.

Gage Factor

The gage factor or strain sensitivity S is defined as

$$S = \frac{\frac{\Delta R}{R}}{\frac{\Delta l}{l}}$$

where Δl is the change in length of any arm and l is its length.

Theoretically, if the conductivity of the wire does not vary with density, it may be calculated that the gage factor will be 2.0 [9]. Actually, measured strain sensitivity factors usually differ from the theoretical by a considerable amount. Calculation of this factor for Nichrome V is made experimentally as follows:

$$\frac{\frac{\Delta l}{l}}{T} = 0.000117 \text{ g.}^{-1},$$

where T is the tension in the wire. (This measurement was necessary due to the large variations in Young's modulus from lot-to-lot of Nichrome V.)

One must reduce $\frac{\Delta R}{R}$ per gram load on the gage to the value corresponding to a 1-g. force in the wire.

From statics, $T = 1.23$ F. Therefore, since $\frac{\Delta R}{R}$ was determined for a 1-g. force on the gage, it must be reduced by a factor of 0.813, so

$$\frac{\frac{\Delta R}{R}}{T} = 0.000325 \text{ g.}^{-1}.$$

From these data the gage factor $S = 2.7$. This may be compared with a value of 2.0 for ordinary Nichrome.

Young's Modulus

It is possible to calculate Young's modulus from the above data, and it is expressed by the equation:

$$M = \frac{Tgl}{\pi r^2 \Delta l} = 23.8 \times 10^6 \text{ p.s.i.},$$

where r is the radius of the wire. The value found is 23.8×10^6 p.s.i., which is in the upper limit of the range as reported by the manufacturer—*viz.*, 12–25 $\times 10^6$ p.s.i.

Gage Load Limits

A 20-g. load on the gage produces a stress in the wire of 69,000 p.s.i. The reported yield stress for this wire is $\cong$ 95,000 p.s.i. Therefore, no larger than 20-g. loads should be applied to a 0.001-in. wire at a 24° sag and for larger forces, larger wire should be employed.

Gage Deflection

The measured gage deflection is approximately 0.004 cm./g. This compares with a value of 0.0053 cm./g. calculated from the gage geometry and the above value of

$$\frac{\frac{\Delta l}{l}}{T}.$$

Thus the stress measuring point is nearly fixed in space.

Deflection Measurements

Since a force may be measured to better than $\pm$ 2 mg. with a gage deflection of 0.004 cm./g., deflections may be measured to approximately $\pm$ 0.000008 cm., $\pm$ 0.08 μ, or $\pm$ 0.0000023 in. (approximately 2 millionths of an inch).

Linearity

It is obviously desirable that the gage voltage output be a linear function of the gage load. In the upper limits of the load range the wire is stretched appreciably, which results in somewhat larger angles of sag. It is evident that as this angle becomes greater, the force amplification decreases. Therefore, as the load increases the gage sensitivity decreases. This variation from linearity does not exceed 0.25% of full scale over all force ranges for sags of 24° or more. Full scale force ranges are

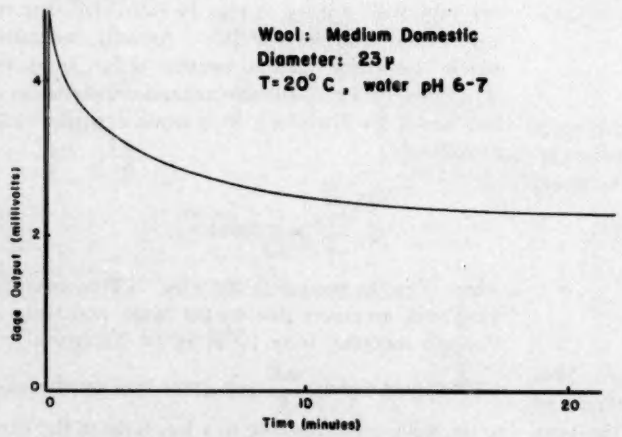

FIG. 7. *Stress-relaxation curve of single wool fiber after 20% elongation in water.*

adjusted by varying the input voltage (see gage equation).

Application of Gage

Its application to the measurement of stress relaxation in wool is illustrated in Figure 7. The fiber was rapidly elongated 20% and held at this length. The gage may also be used in other types of single fiber experiments such as creep, constant rate of elongation, and constant rate of loading.

Summary

The principle and details of construction have been described for a resistance wire strain gage having a number of significant characteristics. These characteristics include: (*1*) high stability, such that under full load there is negligible (better than ½% of full scale) drift or shifting of the zero point and sensitivity over long periods of time (2 months or more). (*2*) high sensitivity, making possible, for example, measurements of force with a precision of 3 mg. or higher. (*3*) gage output sufficient to permit direct coupling to recording potentiometers. (*4*) linearity in response through the full range (up to 20 g. force). (*5*) ruggedness in construction and relative insensitivity to vibration. (*6*) low inertia, allowing millisecond studies on single fibers. (*7*) low gage deflection providing the condition of a stress-measuring point nearly fixed in space.

Acknowledgments

The author wishes to acknowledge the assistance given by Mr. Morris F. Pool and Mr. Andrew Yeiser of this laboratory, and is particularly indebted to Dr. Harold P. Lundgren for his encouragement and suggestions.

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(*Manuscript received November 23, 1953.*)

The Detection of Modification in Animal Fibers

II. Tensile Recovery at 65% R.H.*

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Abstract

Methods are described for measuring parameters of the tensile recovery of animal fibers. The effects of fiber crimp on these measurements, and the interpretation of the data obtained with crimpy fibers, are discussed.

Data are presented on the fiber-to-fiber variation in tensile recovery and on the effects of various chemical treatments.

For wool fibers from a common source there is very little fiber-to-fiber variation in tensile recovery.

Introduction

To detect modification in animal fibers some standardized testing procedure is applied to samples of the untreated and treated fibers. The states of the samples are usually characterized by a few quantitative parameters relating to their response to the testing procedure; the difference between these parameters for untreated and treated samples is taken as a measure of the extent of modification. For example, the Young's modulus and the stress at break of single fibers or fiber bundles, preconditioned in a standardized atmosphere and extended to break using a standardized rate of extension and gage length, could be used as parameters.

The usefulness of any testing procedure depends upon its efficiency in detecting changes in the properties of the fibers which are important from a practical standpoint. For applications where the type of modification is unknown, the ability of the test to discriminate between different types of modification may also be important. The number of samples required to establish a statistically significant difference depends upon the sampling variation for the parameter under consideration, the change in the parameter produced by the degree of modification which it is desired to detect, and the statistical level chosen.

This paper describes a single-fiber testing procedure which yields parameters characteristic of the tensile recovery of animal fibers at 65% R.H. (Tensile recovery is the degree to which stretched fibers are capable of returning to their initial length during and after removal of the stress causing the deformation.) Data are presented illustrating the sampling variation and the changes in the parameters that are produced by various types of chemical modification. The importance, from a practical standpoint, of the tensile recovery behavior of fibers has been discussed by Susich and Backer [6].

Experimental

Measurement of Set and the Effect of Fiber Crimp

Single fibers, mounted with Duco cement between celluloid tabs, were extended at a constant rate of extension using the Instron Tensile Tester [2], and were allowed to contract at the same rate. After a short time at no load the fibers were re-extended. Typical force-extension curves for a straight fiber (human hair) are presented in Figure 1 and for a crimpy fiber (64's grade wool) in Figure 2. The choice of test conditions is discussed below.

The initial uncrimped lengths of the samples were estimated to the nearest 0.01 in. by placing the mounted fibers on a scale with 0.10-in. subdivisions

* This work was conducted as part of the Wool Research Project sponsored by the American Wool Council; the International Wool Secretariat; the National Wool Trade Association; the Office of Naval Research, Department of the Navy; and a number of American wool manufacturing firms. Other phases of this project are sponsored by the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture (RMA Contract A-1s-30934).

Part I of this series appeared in the February, 1951, issue of TEXTILE RESEARCH JOURNAL.

† At present on active duty in the United States Air Force.

and manually extending them until the operator considered the crimp to be just removed.

The parameters of tensile recovery used in this work were the set (unrecovered extension) remaining after the cycle of a given initial extension followed by contraction (set-*1*), and the set remaining after a given recovery time at no load (set-*2*). Initial extension and set were expressed as a percentage of the initial sample length. For straight fibers the measurement of these quantities presented no difficulty; initial extension, set-*1*, and set-*2* were obtained graphically, as shown in Figure 1.

For crimpy fibers (Figure 2), the extension and contraction curves leave and approach the extension axis almost asymptotically. The following assumptions were made to obtain the points *A*, *F*, and *E* at which the curves would depart from and return to the extension axis if crimp had been absent: (*1*) that the linear regions *BC* and *GH* of the extension curves were not greatly affected by the crimp, and that extrapolation of these regions of the curves to the extension axis would yield *A* and *F*, and (*2*) that the force-extension curves for extension-contraction cycles in the uncrimping regions (small extensions up to *B* or *G*) showed little hysteresis, and therefore, that the construction shown in Figure 2 would yield *E*. The re-extension curve was used in this construction because comparison of the force at points *Y* and *X* indicated that the initial extension to *D* had either changed the crimp or weakened the fiber. The parameters of tensile recovery that supposedly would have been measured had crimp been absent could now be obtained as shown in Figure 2. *AE* is set-*1*, *AF* is set-*2*, and *AD* is the initial extension.

A more detailed description of the measurement of the force-extension curves on the Instron Tensile Tester is given in the Appendix (p. 420).

Preparation of Samples

Modified wool lots were prepared by subjecting to various chemical treatments 1-g. to 2-g. samples of an unoiled top made from a U. S. 64's grade wool. Details of the processing into top and of the characteristics of this wool have been reported in a previous publication [*7*]. The chemical treatments are summarized in Table I. Room temperature varied between 20°C and 25°C. The solvent for all reagents was distilled water. After treatment, all lots were washed at room temperature in frequently changed distilled water. A few seconds immersion in ether displaced most of the supernatent water and prevented matting of the fiber mass. The lots were then allowed to dry in an atmosphere at 65% R.H., 70°F before being stored in the dark.

Test Conditions

The tensile recovery of animal fibers depends upon their moisture content (see for example [4]). All measurements described in this paper were made on fibers which had been allowed to come to equilibrium, from the wet side, in a standard atmosphere of 65% R.H., 70°F (controlled to approximately ± 2% R.H. and ± 2°F). (The tensile recovery of wet fibers is discussed in Part III of this paper which immediately follows.)

The measurements of tensile recovery described in this paper are affected by the rates at which the fibers are extended and allowed to contract. A

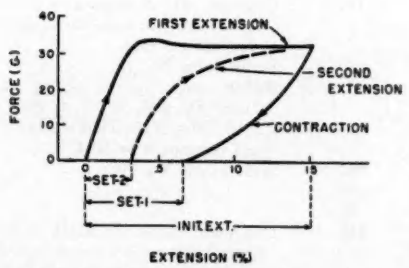

FIG. 1. *Tensile recovery measurement for a straight fiber (human hair). Initial sample length = 1 in., rate of extension = 0.5 in./min., recovery time = 0.06 min., 65% R.H., 70°F.*

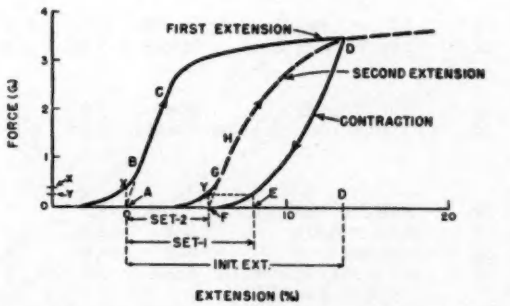

FIG. 2. *Tensile recovery measurement for a crimpy fiber (64's wool). Initial sample length = 1.07 in., rate of extension = 0.5 in./min., recovery time = 0.60 min., 65% R.H., 70°F.*

standard rate of 0.50 in./min. was arbitrarily chosen. The initial sample length of all mounted fibers was approximately 1 in.

The parameters of tensile recovery that were chosen for use in this work were set-*1* and set-*2* for an initial extension to 15% and a recovery time associated with set-*2* of 0.71 to 0.76 min. Data for the dependence of set-*2* on the recovery time, for a given initial extension, are presented in Figure 3. Each measurement was performed with a different fiber from the control lot, and the initial extensions ranged between 14.4% and 15.7%. Variation in the recovery time between 0.71 and 0.76 min. has little effect on the measurement of set-*2*.

Data on the dependence of set on the initial extension are presented in Figure 4. Each pair of set measurements was obtained with a different fiber from the control lot. Due to the experimental procedure, the recovery time associated with set-*2* was dependent on the initial extension (see Appendix, p. 420) and varied from 0.6 min. for 10% initial extension to 1.2 min. for 30% initial extension. For a given initial extension, the recovery time did not vary by more than ± 0.03 min.

Test Procedure and Analysis of Data

The chemically treated wool lots were arranged in three groups, each group being tested on a different date. The test procedure was the same for the three groups.

Fibers were randomly chosen from the control lot and from each of the chemically treated lots in the group being tested. The fibers were mounted on

TABLE I. DETAILS OF CHEMICAL TREATMENTS GIVEN THE WOOL FIBERS

Treatment No.	Reagent	Temperature (°C)*	Time (hr.)	Weight of wool (g.)	Volume of reagent solution (*l*)	Washing time (hr.)	Remarks
1						43	Control
2	Distilled H_2O	Boil	1.0	1	0.8	41	
3	Distilled H_2O	Boil	6.3	1	0.8	45	The treated wool had a yellowish color.
4	9.95% (wt.) $NaHSO_3$	Room	4.5	1	0.5	42	
5	9.95% (wt.) $NaHSO_3$	Room	42.4	1	0.5	95	
6	6 *N* HCl	Room	5.2	1	0.4	41	
7	6 *N* HCl	Room	41.5	1	0.4	94	
8	1.1 *N* NaOH	Room	0.25	1	0.5	91	
9	1.1 *N* NaOH	Room	0.13	1	0.5	91	
10	3% (wt.) H_2O_2	Room	23.8	1	0.5	94	Original pH of reagent = 3.5; ⅙ grain acetanilid per oz. of reagent.
11	1.0% (wt.) $NaHSO_3$	Room	5.0	2	1.0	190	
12	0.50% (wt.) $NaHSO_3$	Room	48.2	2	1.0	205	
13	1.0% (wt.) $NaHSO_3$	50	1.0	2	1.0	70	Original pH of reagent = 4.3.
14	8.8% (wt.) H_2O_2	Room	30.3	2	0.6	194	
15	1% (wt.) H_2O_2	50	12.1	2	0.5	77	Original pH of reagent = 3.8, made from stock solution used for No. 10.
17	3% (wt.) H_2O_2	50	23.0	2	0.5	79	Same as for No. 10.
18	0.01 *N* NaOH	Room	0.25	1	1.0	192	Original pH of reagent = 11.9; wool transferred to a fresh reagent solution after 5 min.
19	0.01 *N* NaOH	Room	1.0	1	1.0	192	Same as for No. 18.
20	$NaHCO_3$–Na_2CO_3 buffer	65	1.0	2	1.0	155	Buffer made by dissolving 6.13 g. $NaHCO_3$ and 29.70 g. Na_2CO_3 in 5.10 liters of distilled water; original pH of reagent = 10.4.
21	$NaHCO_3$–Na_2CO_3 buffer	65	15.5	2	1.0	144	Same as for No. 20.
22	0.011 *N* H_2SO_4	Boil	2.0	1	1.0	191	
26	Distilled H_2O	Boil	12.5	2	1.5	92	
27	0.1 *M* $Hg(CH_3COOH)_2$ in 0.1*N* CH_3COOH	Room	3.5	1	1.0	128	Treatment causes crosslinking [5]; reagent prepared by dissolving 5.72 ml. of glacial acetic acid, and 31.8 g. of mercuric acetate in distilled water; original pH of the reagent = 3.08.
29	$NaHCO_3$–Na_2CO_3 buffer	65	0.17	2	1.0	129	Same as for No. 20.

* Room temperature = 20°–25°C.

celluloid tabs, wet out in water, and conditioned for at least 12 hrs. at 65% R.H., 70°F. The initial lengths of the fiber samples were then measured, and the fibers were tested on the Instron Tensile Tester. In order to average out the effects of slight variations in room conditions, the testing of the fibers from any one lot was spread throughout the entire test period.

For crimpy fibers the point *A* (see Figure 2) cannot be accurately determined until the force-extension curve is available. Therefore, it is impossible to preset the initial extension accurately, and, for a group of fibers nominally extended 15%, the sample-to-sample variation in initial extension lies in the range from 14% to 16%. Since the set depends on the initial extension, this variation in the initial extension has two effects: (*a*) It produces most of the sample-to-sample variation in the measured values of set-*1* and set-*2*. (*b*) The average value of initial extension for the samples from any one lot may not be the same as that for the samples from another lot, and the average values of set will not be directly comparable.

Inspection of Figure 4 shows that both set-*1* and set-*2* are not directly proportional to the initial

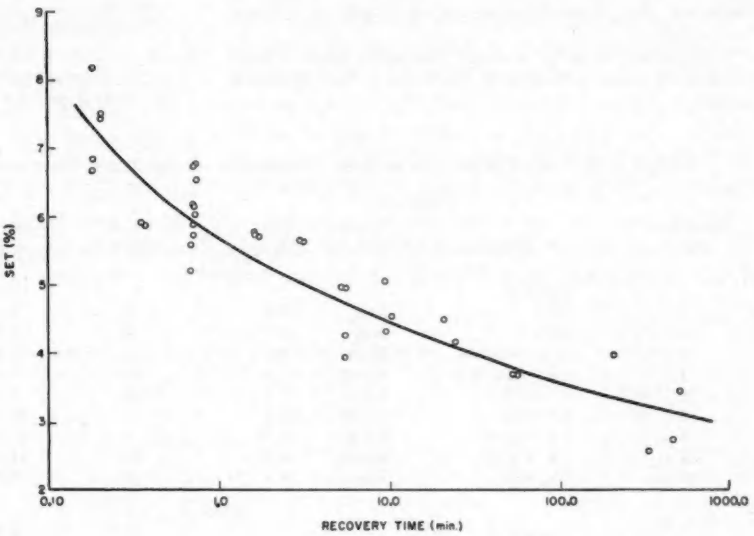

FIG. 3. *Dependence of set-2 on the recovery time of the wool fibers.*

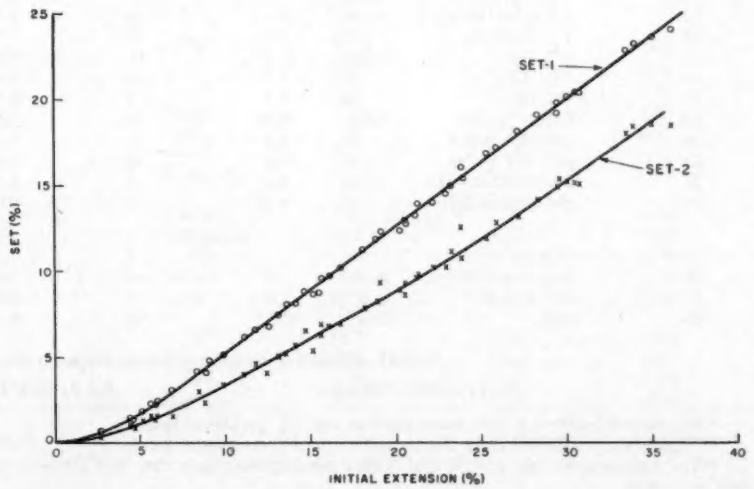

FIG. 4. *Dependence of set on the initial extension of the wool fibers.*

extension.* However, for both set-*1* and set-*2*, the slope of the set *vs.* initial extension curve can be considered to be constant for initial extensions between 10% and 20%, and a least-squares line fitted to the data for the fibers from any one lot provides a method of calculating the mean value of the set for an initial extension to 15%. For both set-*1* and set-*2* these corrected mean values are used to compare the tensile recovery behavior of the different lots. A typical example follows.

Figure 5 is a plot of set-*1* *vs.* initial extension for a typical series of measurements on fibers from the control lot and from one of the treated lots. The bulk of the measurements were made on fibers

* An attempt to derive a simple parameter which is independent of initial extension is described in the Appendix, p. 420.

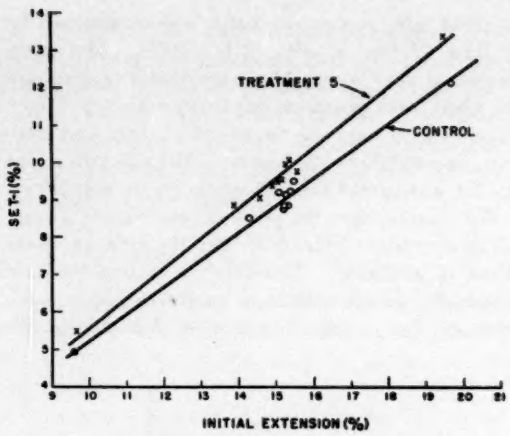

FIG. 5. *Comparison of the set-1 measurements for the control and for a treated lot of the wool fibers.*

TABLE II. THE EFFECT OF CHEMICAL TREATMENT OF THE WOOL FIBERS ON THEIR TENSILE RECOVERY

Treatment No.	Treatment: Reagent	Temp. (°C)	Time (hr.)	No. of fibers	Set-*1* (%)	Set-*2* (%)	(Set-*1*)–(Set-*2*) (%)
			Group 1				
1	Control			17	8.85	6.18	2.7
2	H_2O	Boil	1.0	11	8.95	6.10	2.8
3	H_2O	Boil	6.3	9	9.23**	6.51*	2.7
4	10% $NaHSO_3$	Room	4.5	13	9.23**	6.71**	2.5
5	10% $NaHSO_3$	Room	42.4	13	9.58**	7.03**	2.6
6	6*N* HCl	Room	5.2	14	9.15**	6.35	2.8
7	6*N* HCl	Room	41.5	6	10.17**	7.93**	2.2
8	1*N* NaOH	Room	0.25	5	11.20**	9.39**	1.8
9	1*N* NaOH	Room	0.13	10	11.06**	8.84**	2.2
10	3% H_2O_2	Room	23.8	14	9.20**	6.65**	2.6
			Group 2				
1	Control			10	8.96	6.21	2.8
11	1% $NaHSO_3$	Room	5.0	10	9.09	6.34	2.8
12	0.5% $NaHSO_3$	Room	48.2	10	9.24**	6.71**	2.5
13	1% $NaHSO_3$	50	1.0	10	9.27**	6.61*	2.7
14	9% H_2O_2	Room	30.3	10	9.64**	7.12**	2.5
15	1% H_2O_2	50	12.1	10	9.44**	6.80**	2.6
17	3% H_2O_2	50	23.0	6	10.51**	8.63**	1.9
18	0.01*N* NaOH	Room	0.25	10	9.04	6.29	2.8
20	pH 10.4 buffer	65	1.0	10	9.14	6.44	2.7
21	pH 10.4 buffer	65	15.5	10	9.44**	6.78**	2.7
27	Mercuric acetate	Room	3.5	10	8.89	6.83**	2.1
29	pH 10.4 buffer	65	0.17	10	9.06	6.37	2.7
			Group 3				
1	Control			5	9.15	6.44	2.7
19	0.01*N* NaOH	Room	1.0	10	9.06	6.36	2.7
22	0.01*N* H_2SO_4	Boil	2.0	7	9.33	6.81	2.5
26	H_2O	Boil	12.5	10	9.57**	7.00**	2.6

Pooled‡ variances of deviations from least-squares line:

Set-*1*: 0.0492 (188 d.f.) Set-*2*: 0.1454 (188 d.f.)

* Significantly different from the control at the 5% confidence level.

** Significantly different from the control at the 1% confidence level.

‡ The variances for lots 2, 7, 8, and 9 were not included since they had not been calculated at the time the pooled estimate was made.

nominally extended to 15% initial extension; in order to establish the regression coefficients with a fair degree of accuracy, a few fibers from each lot were usually extended to 10% or 20% initial extension. The least-squares lines are shown, and the mean values of the set at 15% initial extension can be calculated from the equations of the lines. The variances of these means can be obtained from the deviations of the individual measurements from the least-squares lines, and the statistical significance of the difference between the means can be evaluated.

Results and Discussion

The results of the measurements of tensile recovery are summarized in Table II. The values of set-*1* and set-*2* have been corrected to a 15% initial extension, and their averages are reported. For either set-*1* or set-*2*, the variances were homogeneous among the various lots; therefore, only the pooled variance is reported. The Student *t*-test [3] was used to estimate the statistical significance of the difference between the mean for any treated lot and the mean for the control lot of the group in which the treated lot was tested. Subdivision of the lots into three groups was preserved because of the possibility of the average atmospheric conditions being slightly different on the three testing dates. In Table III, the means for the treated lots have been arranged according to type of treatment, and they are expressed relative to the mean for the control lot of the group in which the treated lot was tested.

Within any one lot, there was very little fiber-to-fiber variation in tensile recovery. For fibers from the control lot the mean for set-*1* corrected to 15% initial extension was 8.93; the standard deviation of individuals from the least-squares line was only 0.22. For set-*2* the mean was 6.23 and the standard deviation was 0.37. Much of this fiber-to-fiber variation may be due to slight fluctuations in atmospheric conditions during the testing of any one group. It can be concluded that the tensile recovery behavior of wool fibers from a common source is very uniform.

All of the chemical treatments, which significantly affected the tensile recovery, caused increases in set-*1* and set-*2*. The difference between set-*1* and

TABLE III. THE EFFECT OF CHEMICAL TREATMENT OF THE WOOL FIBERS ON THEIR TENSILE RECOVERY

Treatment No.	Reagent	Temp. (°C)	Time (hr.)	Relative* set-*1*	Relative* set-*2*
2	H_2O	Boil	1.0	1.01	0.99
3	H_2O	Boil	6.3	1.04	1.05
26	H_2O	Boil	12.5	1.05	1.09
22	0.01*N* H_2SO_4	Boil	2.0	1.02	1.06
6	6*N* HCl	Room	5.2	1.03	1.03
7	6*N* HCl	Room	41.5	1.15	1.28
29	pH 10.4 buffer	65	0.17	1.01	1.02
20	pH 10.4 buffer	65	1.0	1.02	1.04
21	pH 10.4 buffer	65	15.5	1.05	1.09
18	0.01*N* NaOH	Room	0.25	1.01	1.01
19	0.01*N* NaOH	Room	1.0	0.99	0.99
9	1*N* NaOH	Room	0.13	1.25	1.43
8	1*N* NaOH	Room	0.25	1.27	1.52
11	1% $NaHSO_3$	Room	5.0	1.01	1.02
12	0.5% $NaHSO_3$	Room	48.2	1.03	1.08
13	1% $NaHSO_3$	50	1.0	1.03	1.06
4	10% $NaHSO_3$	Room	4.5	1.04	1.09
5	10% $NaHSO_3$	Room	42.4	1.08	1.14
15	1% H_2O_2	50	12.1	1.05	1.09
10	3% H_2O_2	Room	23.8	1.04	1.08
17	3% H_2O_2	50	23.0	1.17	1.39
14	9% H_2O_2	Room	30.3	1.07	1.14
27	Mercuric acetate	Room	3.5	0.99	1.10

* Relative to the control lot.

set-*2* is a rough measure of the rate of delayed recovery, and set-*1* minus set-*2* is tabulated in the last column of Table II. In general, the chemical treatments tended to decrease the rate of delayed recovery, and for a given modification the percentage change in set-*2* is greater than that in set-*1*. In one case, the crosslinking with mercuric acetate, set-*1* was not affected by the treatment whereas set-*2* was increased. In most cases, however, the greater fiber-to-fiber variation in set-*2* more than compensated for the greater change in this parameter, so that set-*2* was less efficient in detecting modification than was set-*1*.

Even though fiber-to-fiber variation is small for both parameters, it is less for set-*1* than for set-*2*. Several investigators (see summary in [6]) have discussed the necessity of distinguishing between immediate and delayed tensile recovery processes. Since set-*2* is influenced by the delayed recovery processes to a much greater extent than is set-*1*, it is possible to conclude that, for wool fibers from a common source, the fiber-to-fiber variation in the immediate recovery process is less than that in the delayed recovery processes.

Summary and Conclusions

A method has been described for measuring the following parameters of the tensile recovery of animal fibers: Set-*1*.—The set remaining after a cycle of extension to 15% at a constant rate of extension and contraction at the same rate. Set-*2*.—The set remaining after a further short recovery time.

The effects of fiber crimp on these measurements and the interpretation of the data obtained with crimpy fibers are discussed.

Data are presented on the dependence of set on initial extension and on recovery time.

The fiber-to-fiber variation in tensile recovery and the effects of a variety of chemical modifications have been studied. These data can be used to estimate how many fibers must be tested for the detection of a given amount of chemical modification. For most types of modification, set-*1* is the more efficient parameter.

For wool fibers from a common source there is very little fiber-to-fiber variation in tensile recovery.

Acknowledgment

The author wishes to thank Professor J. C. Whitwell for his help in clarifying the presentation, and Miss Edith Voong for mounting many of the fibers.

Appendix

Measurement of Tensile Recovery on the Instron Tensile Tester

Some familiarity with the operation of the Instron Tensile Tester [2] will be assumed. The fiber is mounted between the load-sensing element and the movable crosshead (Figure 6). For this experiment, the load-sensing element may be considered to be immovable. Downward motion of the crosshead causes extension of the fiber; upward motion allows it to contract. The force-extension curve, traced on the recorder chart, for a typical measurement of set-*1* and set-*2* is given in Figure 7.

By manually lowering the crosshead, a slight pretensioning force is applied to the fiber. The Instron controls are set to cause the crosshead to cycle between the upper reversal point (a short distance above the pretensioning point) and the lower reversal point (a distance below the pretensioning point equal to the desired extension); and the crosshead is started moving up. The Instron controls are further set so as to cause a pip to be registered on the chart when the crosshead reaches the upper reversal point. The crosshead is allowed to move (at a constant rate of motion) between the two reversal points until the force-extension curves shown in Figure 7 have been obtained.

At 0.5 in./min. rate of motion of the crosshead, the unrecovered time lag during reversal of direction of motion is negligible. The quantities l_1, l_2, l_3, and e are measured from the force-extension curves, and

$$\text{set-}1 = 100\,\frac{l_2 - l_1}{\text{initial length}},$$

$$\text{set-}2 = 100\,\frac{l_3 - l_1}{\text{initial length}},$$

$$\text{initial extension} = 100\,\frac{e}{\text{initial length}}.$$

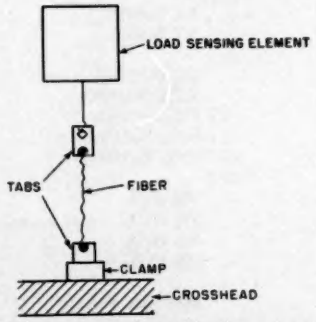

FIG. 6. *Schematic diagram of a fiber mounted in the tensile tester.*

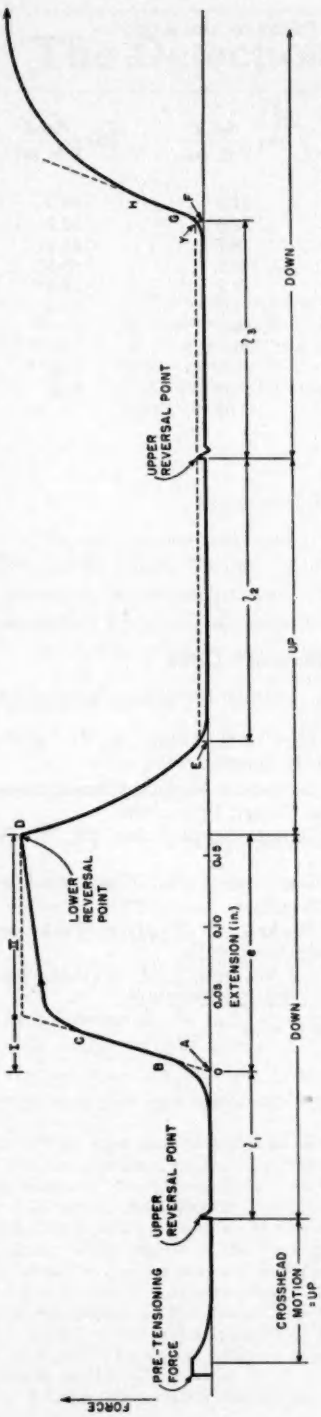

FIG. 7. *Typical force-extension curves for a tensile recovery measurement on crimpy wool fibers. Initial sample length = 1.09 in., rate of extension = 0.5 in./min., 65% R.H., 70°F.*

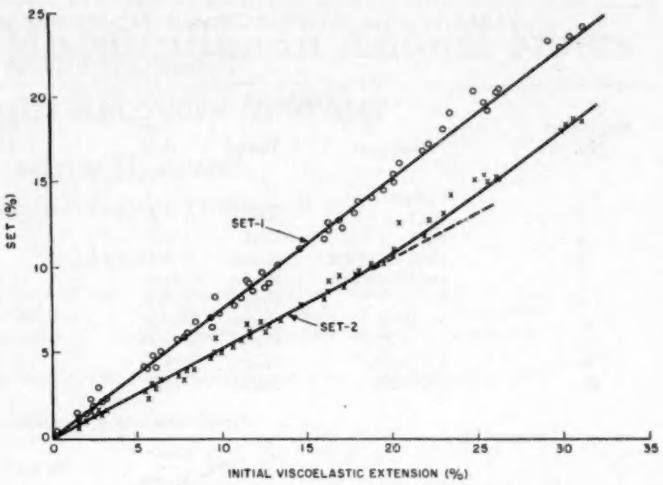

FIG. 8. *Dependence of set on initial viscoelastic extension of the wool fibers.*

Since the recovery time associated with set-*2* is the interval between points *E* and *F*, it is, with this procedure, dependent on the preset values of *e* and l_1.

Parameters Independent of the Initial Extension

It would be useful to have a parameter of tensile recovery behavior that does not depend on the initial extension. If the slope of the force-extension curve (Figure 7) in the region *B-C* is taken as a measure of the instantaneous elastic modulus [1], the total initial extension can be divided into two parts: (*1*) a measure of the instantaneous elastic extension (primary creep), and (*2*) a measure of the viscoelastic extension (secondary creep and permanent set).

In Figure 8, set-*1* and set-*2* are plotted against the initial viscoelastic extensions. Each pair of set measurements was obtained with a different fiber from the control lot. Inspection of Figure 8 shows that the ratio of set-*1* to initial viscoelastic extension was independent of initial extension over the entire range studied (up to 35% initial total extension). The ratio of set-*2* to initial viscoelastic extension was independent of initial extension up to an initial total extension of 20%.

These ratios have been measured from the experimental data for the fibers tested in *Group I* that had initial extensions to approximately 15%, and the

TABLE IV. THE EFFECT OF CHEMICAL TREATMENT OF THE WOOL FIBERS ON THE RATIO $100 \frac{\text{SET}}{\text{VISCOELASTIC EXTENSION}}$

Treatment No.	Treatment: Reagent	Temp.	Time (hr.)	No. of fibers	$100 \frac{\text{Set-}1}{\text{V.E. ext.}}$	$100 \frac{\text{Set-}2}{\text{V.E. ext.}}$
			Group 1			
1	Control			10	77.5	53.3
2	H_2O	Boil	1.0	10	78.0	52.7
3	H_2O	Boil	6.3	9	78.7	55.4
4	10% $NaHSO_3$	Room	4.5	10	78.3	56.6*
5	10% $NaHSO_3$	Room	42.4	10	79.5	58.2**
6	6*N* HCl	Room	5.2	10	77.7	53.6
7	6*N* HCl	Room	41.5	6	86.5**	67.4**
8	1*N* NaOH	Room	0.25	5	89.7**	75.3**
9	1*N* NaOH	Room	0.13	10	87.8**	71.4**
10	3% H_2O_2	Room	23.8	10	77.0	55.6

Pooled variances of individuals:

$100 \frac{\text{Set-}1}{\text{V.E. ext.}}$: 12.5 (80 d.f.)

$100 \frac{\text{Set-}2}{\text{V.E. ext.}}$: 13.7 (80 d.f.)

* Significantly different from the control at the 5% confidence level.
** Significantly different from the control at the 1% confidence level.

results are summarized in Table IV. The values for the individual fibers did not have to be corrected for the slight variations in initial extension. However, chemical modification increased that portion of the total extension which was measured as the viscoelastic extension, as well as the set. Therefore, for a given degree of chemical modification the percentage change in the ratio $\frac{\text{set}}{(\text{viscoelastic extension})}$ tended to be less than the percentage change in set alone, and the use of the ratio was not as efficient for detecting modification as was the method described in the main text of this paper.

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(Manuscript received October 2, 1953.)

The Detection of Modification in Animal Fibers

III. Tensile Recovery in Water*

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Abstract

The tensile recovery of animal fibers in water has been measured in a manner similar to that previously described [1] for animal fibers at 65% R.H. The tensile recovery measured in water was much less sensitive for detecting chemical modification than the tensile recovery at 65% R.H.

Even after very severe chemical modification, water-soaked animal fibers showed almost perfect tensile recovery.

Introduction

It is well known that animal fibers extended and allowed to contract while immersed in water show almost perfect tensile recovery. However, very small amounts of set can be measured by using the techniques developed in the preceding paper in this series [1], and it is possible to obtain parameters characteristic of the tensile recovery of wet animal fibers. As in the preceding paper, the fiber-to-fiber variability of these parameters, and the effects of chemical treatment on these parameters, have been determined.

Experimental

The tensile recovery (of fibers immersed in distilled water at 70°F) was measured in a manner similar to that described in Part II of this series. Although these fibers were allowed to soak in water for several hours prior to testing, separate experiments showed that, insofar as the measurement of tensile recovery was concerned, the fibers reached equilibrium with the water within 1.5 min. The parameters of tensile recovery that were used were set-*1*, the set remaining after a given amount of extension at a constant rate followed by contraction at the same rate, and set-*2*, the set remaining after a short additional recovery time. The rate of extension was 0.1 in./min.; the initial sample length was approximately 1 in.

Data on the relationships between set and initial extension are presented in Figures 1 and 2. In either figure, each point represents a measurement on a

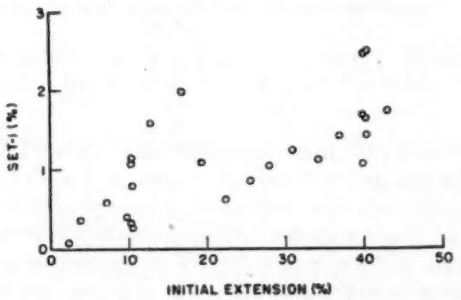

FIG. 1. *Dependence of set-1 on the initial extension of the wool fibers.*

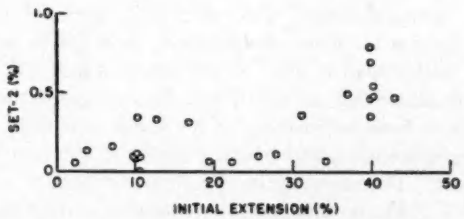

FIG. 2. *Dependence of set-2 on the initial extension of the wool fibers.*

* This work was conducted as part of the Wool Research Project sponsored by the American Wool Council; the International Wool Secretariat; the National Wool Trade Association; the Office of Naval Research, Department of the Navy; and by a number of American wool manufacturing firms. Other phases of the Project were sponsored by the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture (RMA Contract A-1s-30934).

Part I of this series appeared in the February, 1951, issue of TEXTILE RESEARCH JOURNAL and Part II appears in this issue, p. 414.

† At present on active duty in the United States Air Force.

TABLE I. THE EFFECT OF CHEMICAL TREATMENT OF THE WOOL FIBERS ON THEIR TENSILE RECOVERY (INITIAL EXTENSION = 30%)

Treatment No.	Treatment: Reagent	Temp.	Time (hr.)	No. of fibers	Set-*1* (%)	Set-*2* (%)
1	Control			10	1.13	0.18
3	H_2O	Boil	6.3	8	1.17	0.14
4	10% $NaHSO_3$	Room	4.5	10	1.42	0.23
5	10% $NaHSO_3$	Room	42.4	10	1.12	0.15
9	1*N* NaOH	Room	0.13	13	1.05	0.28
10	3% H_2O_2	Room	23.8	8	0.70	0.11

Pooled variances of individuals:

Set-*1*: 0.154 (53 d.f.)
Set-*2*: 0.0135 (53 d.f.)

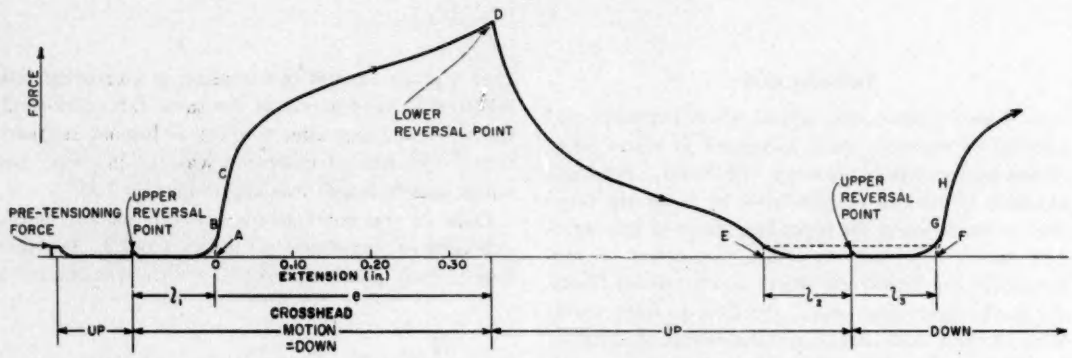

FIG. 3. *Typical force-extension curves for a tensile recovery measurement on crimpy wool fibers. Initial sample length = 1.18 in. Rate of extension = 0.5 in./min. Recovery time = 0.44 min. Distilled water at 70°F.*

different fiber from the control lot of the 64's wool. The recovery time associated with set-*2* varied only slightly with initial extension; it averaged about 0.43 min. Since a set of 0.5% represents a change in the length of the fiber of only 0.005 in., the scatter of the points is not surprising. For most of the work described in the paper the initial extension was standardized at 28%–32%, and the recovery time associated with set-*2* at 0.42–0.46 min.

As was described in the preceding paper, the initial extension for these crimpy fibers could not be preset to better than ± 1%. Inspection of Figures 1 and 2 indicates that for wet fibers, the variance in the set due to such inconstancy of the initial extension was negligible compared to other errors in the measurement. Therefore, unlike the measurements of set at 65% R.H., no attempts were made to correct variations in the initial extension. All fibers were extended to the approximate preset extension.

Fibers were tested from the control and from several of the chemically treated wool lots described in the preceding paper. With the exceptions described above, the testing procedure was the same as that used in the preceding paper. The force-extension curves obtained for a typical measurement on a fiber from the control lot are presented in Figure 3. (Compare with Figure 7 of the preceding paper.)

Results and Discussion

The results of measurements of the tensile recovery of the wet fibers after a 30% initial extension are summarized in Table I. For one of the treated wool lots the average breaking extension was less than 30%, and this lot was compared with the control using an initial extension of 10%; the results of these measurements are summarized in Table II.

As was pointed out above, the experimental error for the measurement of the set was quite high. For

TABLE II. THE EFFECT OF CHEMICAL TREATMENT OF THE WOOL FIBERS ON THEIR TENSILE RECOVERY (INITIAL EXTENSION = 10%)

Treatment No.	Treatment Reagent	Temp.	Time (hr.)	No. of fibers	Set-*1* (%)	Set-*2* (%)
1	Control			4	0.35	0.00
7	6*N* HCl	Room	41.5	5	1.12**	0.03

Pooled variances of individuals:

Set-*1*: 0.0455 (7 d.f.)
Set-*2*: 0.00039 (7 d.f.)

** Significantly different from the control at the 1% confidence level.

a 30% initial extension the coefficient of variation for set-*1* was 35%; for set-*2* it was 64%.

The statistical significances of the differences between the mean value of set-*1* or set-*2* for a treated lot, and set-*1* or set-*2* for the control lot have been evaluated. Set-*1* for treatment 7 is significantly high (1% confidence level); set-*2* for treatment 7 and set-*2* for treatment 9 may be high (10% confidence level). For measurements at 65% R.H., however, both set-*1* and set-*2* for all of these treated lots were significantly higher than the set-*1* and set-*2* values for the control. Therefore, for detecting chemical modification, tensile recovery measured in water was much less sensitive than tensile recovery measured at 65% R.H.

It is of interest to note that, even after very severe chemical treatments such as treatments 7 or 9, the wet fibers showed almost perfect tensile recovery. Similarly, Harris, Mizell, and Fourt [2] have shown that wet wool fibers show complete tensile recovery even after five-sixths of the cystine cross-links have been broken.

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(*Manuscript received October 2, 1953.*)

Correction

In the article "Irregularity in Textile Yarns and Strands: A Literature Survey," by Malcolm A. Sanborn, which appeared in the January, 1954, issue of TEXTILE RESEARCH JOURNAL, the Enrick formula for expected coefficient of variation (p. 90) should read:

"$R = 0.816 \sqrt{(\text{yarn number})\ (\text{fiber fineness } (\mu\text{g./in.}))}$."

Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

Static Electrification of Filaments

TEXTILE RESEARCH INSTITUTE
Princeton, New Jersey
February 12, 1954

To the Editor
Textile Research Journal

Dear Sir:

The earliest systematic quantitative study of frictional electrification between solids was made by Péclet [3], who studied the influence on electrification of velocity, pressure, extent of surface of contact, state of surface, thickness of bodies, nature of materials, and in a later paper [4] the effect of carbon dioxide instead of air for the surrounding atmosphere. Among subsequent investigations an outstanding work is that of Macky [2], who with a much more refined apparatus confirmed and extended Péclet's work. One of Macky's important results was that the net amount of charge transferred decreases with decreasing atmospheric pressure.

In the works cited, as in others of those periods, the researches were carried out with rather large samples, and a considerable area of one material was rubbed against a correspondingly large area of another material. We have hoped to go a step further in elucidating the complex phenomena of static electrification by using smaller areas of contact in order to obtain increased homogeneity of material and better defined geometry. Our experiments consisted of measuring the amount of charge produced when one filament of a given material is rubbed against another of the same or different material, under controlled mechanical and ambient conditions. Among the materials studied were the metals gold, platinum, iron, aluminum, magnesium, and copper, and the nonmetals glass, wool, cotton, nylon, acetate, viscose, polyethylene, and Orlon. A filament of one of these substances is placed in an apparatus in which one fiber is held fixed while a second fiber is rubbed across it and then removed essentially to infinity. The transferred charge (10–1000$\mu\mu$c.) left on the stationary fiber is determined by measuring with a vacuum-tube electrometer the potential change (0.1–10v.) on the system of known capacitance (20–200$\mu\mu$f.). A radium source was used to neutralize the charge when desired. The fibers were shielded by Faraday cages, and the whole apparatus was contained in a chamber at controlled temperature and humidity.

The reproducibility of the experimental results was good, a finding in conformity with the earlier works cited. Repetition of a rub between a given pair of filaments gave a value for charge generated constant within 5% or better, after a number of preliminary rubs ranging from two or three for nylon on polyethylene to sixty or more for nylon on acetate. The reproducibility from one pair of filaments of given materials to another pair was moderately good, the final value of charge ordinarily varying not more than 20% from the mean. The sign of charge generated between two types of filament was quite consistent and permitted establishment of a triboelectric series largely in conformity with those of other investigators. (See for example [1].)

The effect of temperature was studied over only a very small range, 25°C to 30°C, where it was established that there exists no strong dependence which might have masked other effects at this stage of the investigation. The effect of relative humidity was pronounced, the net charge transferred being small at high humidities. The effects of other changes in atmosphere—composition or pressure—have not yet been studied.

The effects of mechanical parameters were studied at 30°C and 33% relative humidity on many, but not all, combinations of the materials listed earlier. Our results may reasonably be described by the following generalizations:

Metals on insulators.—The amount of charge generated is: (*a*) proportional to length of stroke; (*b*) linearly increasing with normal force, until a limiting value is reached with some materials, but not with others. The intercept of the curve at vanishing force does not vanish; (*c*) independent of tension on the filaments; (*d*) dependent on manner of rub,* in a way not easily summarized; (*e*) linearly increasing with velocity until a limiting value is reached. The intercept of the curve at vanishing velocity does not vanish.

Insulators on insulators.—The amount of charge generated is: (*a–d*) as above; (*e*) independent of velocity.

Metals on metals.—The amount of charge generated is independent of mechanical parameters, mere contact sufficing to transfer an amount apparently determined by the capacitance of the system and the contact potential between the metals.

A generalization concerning the amount of charge transferred and the position of the materials in the triboelectric series is that the amount seems to be largely independent of position in the series for insulators on insulators, whereas for metals on insulators the amount seems to depend on the position of the insulator in the series and the photoelectric work function of the metal.

Thus far, we have been unable to postulate a quantitative theory to describe all these effects. Concerning the sign of charge transferred, the position in the triboelectric series seems related to the ionization potential of monomers of the artificial polymers, a high position corresponding to a low ionization potential. But electron affinity would seem to be at least as significant a factor. At present we do not have adequate data to study its influence. Concerning the amount of charge transferred, it seems well to wait until additional data are available on the effect of ambient pressure and on the effect of temperature over wide ranges. Moreover, it seems likely that different mechanisms may apply for metals on insulators and for insulators on insulators.

An account of the details of this work and its extension, together with some theoretical speculations, will appear later. Gratitude is expressed for the support of this work by the Carbide and Carbon Chemicals Fellowship at Textile Research Institute.

* This refers to the orientation of the axes of the filaments with respect to the direction of their relative motion.

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INDUSTRIAL SECTION

Effect of Single and Ply Twists on the Properties of a 15.5/2 Carded Cotton Yarn

Louis A. Fiori, John J. Brown, and Jack E. Sands

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Abstract

The effect of single and ply twists on the properties of a 15.5/2 carded yarn was studied, and a comparison made with the results of a similar study conducted on a 31/2 carded cotton yarn. In addition, the properties of a single yarn (15.5/1) were compared to those of an equivalent 2-ply yarn (31/2). Generally, the ply twist-strength curves for the 15.5/2 yarn were found to be similar to those for the 31/2 yarn. However, a change to a coarser yarn number caused definite differences in the twist level required for maximum strength and in the characteristics of the maximum strength plateaus. The elongation characteristics of the 15.5/2 and 31/2 yarns were also very similar, but the elongation values for the coarser yarns were higher than for the finer yarns. With decreasing yarn number, low single twist yarns reacted less sensitively elongationwise to high ply twist. For equivalent single and ply twist multiplier combinations, maximum ply yarn strength values were obtained with approximately the same twist combination for both the 15.5/2 and 31/2 yarns; elongation values increased as equivalent twist multiplier combinations increased, with the rate of increase being greater for the coarser yarn. For low and medium twist single yarns the percentage gain of ply over single yarn strength became less as the yarn became coarser. The equivalent ply yarn (31/2) was generally stronger and was, in most cases, lower in elongation than its single yarn counterpart (15.5/1). Ply yarn variability seemed to increase as the twist increased in the component single yarns.

IN a previous report† the authors discussed the effect of single and ply yarn twists on the the properties of a 31/2 yarn. Although yarn number was not evaluated as a variable in that study, it was expected that a change in yarn number would have influenced the findings to some degree. Therefore, a study has been made with the same cotton and processing organization, but using a coarser yarn number. Reported here are properties of a 15.5/2 yarn and a discussion relating the properties of a single yarn (15.5/1) to those of an equivalent 2-ply yarn (31/2). In order to understand fully the discussions presented in this report, it will be necessary to refer to the report on the 31/2 yarn.

To facilitate comparisons of comparable data, figures for the two reports are numbered similarly.

Procedures and Methods

The cotton, machines, processing organization (except for twist multiplier series), and tests for determining physical properties of fibers and yarns in this study were identical to those previously reported.† Briefly, the cotton was a Deltapine of average staple length, fineness, and grade; machines, a Whitin Casablanca‡ long-draft spinning frame (5-in. gage) and twister (4½-in. gage); and twist multiplier organization, for single yarns, from 2.9 through 6.9 in increments of one, and for ply yarns, from 1.9 through 7.9 in increments of two. In addition, a "balancing" ply twist multiplier was

* One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Service, U. S. Department of Agriculture.

† Fiori, L. A., Brown, J. J., and Sands, J. E., Effect of Single and Ply Twists on the Properties of a 31/2 Carded Cotton Yarn, TEXTILE RESEARCH JOURNAL 24, 267–72 (1954).

‡ Mention of trade names does not imply their endorsement by the Department of Agriculture or TEXTILE RESEARCH JOURNAL over similar products not mentioned.

combined with each single yarn twist multiplier construction. The "balanced" characteristic of the yarn was determined immediately after twisting by taking the yarn off the side of the bobbin and then observing whether a loop of yarn folded or twisted on itself. It is recognized that the "balanced" characteristic of a yarn is dependent on local processing conditions and the twist construction used to produce it and that it is subject to small adjustments.

Analysis of Data

Mainly, comparison is made between the ply yarn strength and elongation data of this study (15.5/2) and comparable data of the study previously reported. Analyses and comparisons were made from six standpoints: (*1*) varying ply yarn twist, with single yarn twist constant; (*2*) varying single yarn twist, with ply yarn twist constant; (*3*) equivalent single and ply yarn twist multiplier combinations; (*4*) ply-single yarn strength ratios of the 15.5/2 yarns; (*5*) equivalent yarn number comparison of a 15.5/1 yarn with a 31/2 yarn; and (*6*) variability of the ply yarns.

Effect of Ply Yarn Twists

Strength Data

Figure 1 shows curves which relate ply yarn twists to skein strength when the twist in the single yarn is held constant. Generally, the ply twist-strength curves for the 15.5/2 yarn are similar to those for the 31/2 yarn. However, the change to a coarser yarn caused definite differences in the twist level required for maximum strength and in the characteristics of the maximum strength plateaus. Of the single yarn twist multipliers used, only the 3.9 and 5.9 twist multiplier yarns show points of maximum strength, whereas with the 31/2 yarns all single-ply twist combinations produced points of maximum strength. As compared with the 31/2 yarns, the twist required for maximum strength of the 15.5/2 yarns is at a higher level with the 2.9 single yarn twist multiplier and at a lower level with all other twist multipliers. Maximum strength plateaus of 15.5/2 yarns are relatively broader, permitting larger changes in ply twist (approximately 2 twist multipliers) without materially decreasing yarn strength.

An area of approximately the same yarn strengths is apparent with the 15.5/2 yarn with single yarn twist multiplier constructions between 2.9 and 5.9 and ply twist multipliers between 3.9 and 4.4 (approximately). The range of single twist multipliers for this area of equal strengths is greater for the 15.5/2 than for the 31/2 yarns.

The strongest "balanced" yarns were spun using a 4.9 single yarn twist multiplier as a basis. Incidentally, in this study, the "balancing" ply twist multipliers were found to be approximately 0.7 of the single yarn twist multipliers.

Figure 2 shows curves which relate ply yarn twist to single strand strength when the twist in the single yarn is held constant. Generally, these curves resemble those based on skein strength values (Figure 1). One exception noted was that the area of approximately the same strengths was at a higher ply twist (approximately 5.9 T.M.) level for single strand strength than for skein strength (Figure 1).

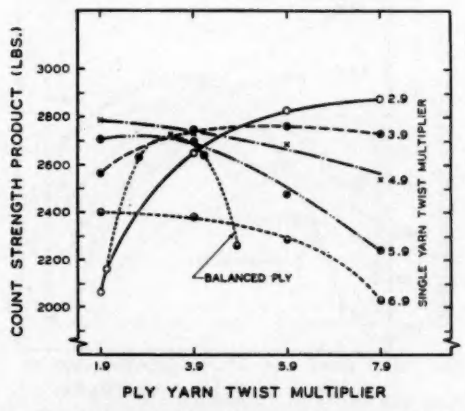

FIG. 1. *Effect of ply twist on skein strength of a 15.5/2 carded cotton yarn.*

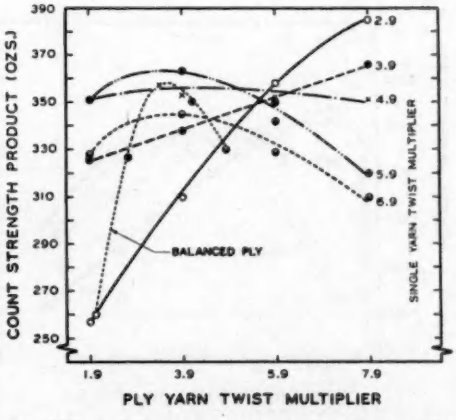

FIG. 2. *Effect of ply twist on single strand strength of a 15.5/2 carded cotton yarn.*

The wide strength plateau of the 4.9 single yarn twist multiplier curve is of particular importance since increases over the full range of ply twists (1.9 to 7.9 T.M.) cause only small changes in yarn strength. This single yarn twist multiplier (4.9) is the same as the one required to obtain maximum single yarn single strand strength (see Figure 9B).

A comparison of the 15.5/2 and 31/2 data shows remarkable similarity in trends and in count strength product values for given combinations of single and ply twists.

The strength trend of the "balanced" construction was similar to that of the skein values. Here, also, the strongest "balanced" yarn was produced from the single yarn spun with a 4.9 twist multiplier.

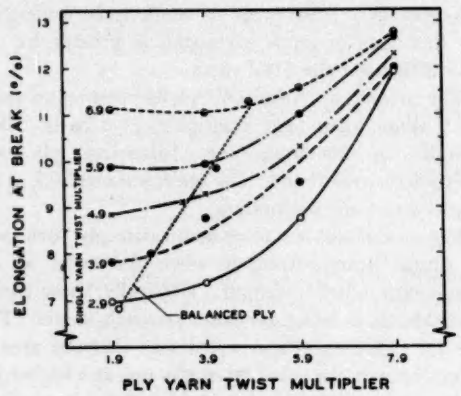

FIG. 3. *Effect of ply twist on elongation of a 15.5/2 carded cotton yarn.*

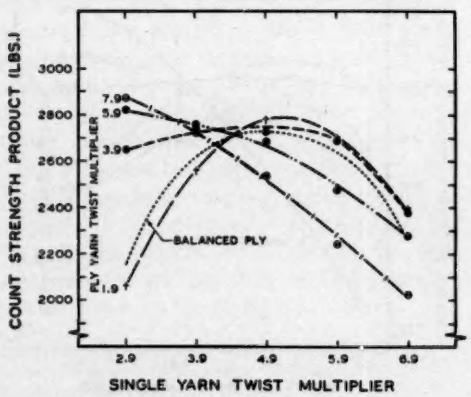

FIG. 4. *Effect of single yarn twist on skein strength of a 15.5/2 carded cotton yarn.*

Elongation Data

Figure 3 presents curves which relate ply yarn twist to elongation at break, when the twist in the single yarn is held constant. The elongation characteristics of the 15.5/2 and 31/2 yarns are very similar; however, yarn number does influence these relationships. For instance, elongation values for the 15.5/2 yarns, regardless of single-ply twist combinations, are higher than for the 31/2 yarns.

Maximum elongation was obtained with a high single (6.9) in combination with a high ply (6.9) twist multiplier for the 15.5/2 yarn. In contrast, the twist multiplier combination which produced maximum elongation for the 31/2 yarn was a low single (2.9) in combination with a high ply (7.9) twist multiplier.

The characteristics of the 15.5/2 and 31/2 twist-elongation curves are generally similar except that for the former yarn none of the curves cross each other. Therefore, with decreasing yarn number low single twist yarns react less sensitively elongationwise to high ply twist.

Effect of Single Yarn Twist

Strength Data

Figures 4 and 5 show curves which relate single yarn twist to ply skein strength and single strand strength when ply twist is held constant. These curves display approximately the same trends as those of the 31/2 yarns. The basic difference is that as the yarn becomes coarser, the area of approximately equal strengths becomes better defined and

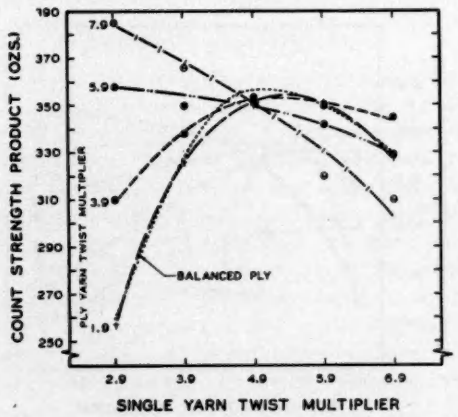

FIG. 5. *Effect of single yarn twist on single strand strength of a 15.5/2 carded cotton yarn.*

includes a larger range of single and ply twist multipliers.

Elongation Data

Figure 6 shows curves which relate single yarn twist to elongation at break when the twist in the ply is held constant. Yarn number influences elongation significantly, since the rate of elongation increase for all twist combinations is greater for the 15.5/2 than for the 31/2 yarns.

It should be noted that the "balanced" ply curves of Figures 4, 5, and 6 are plotted only with reference to the single yarn twist multipliers in combination with "balancing" ply twist and the count strength product. Since the "balancing" ply yarn twist multipliers increase with increasing single yarn twist multipliers, comparisons with the other constructions can only be made on this basis.

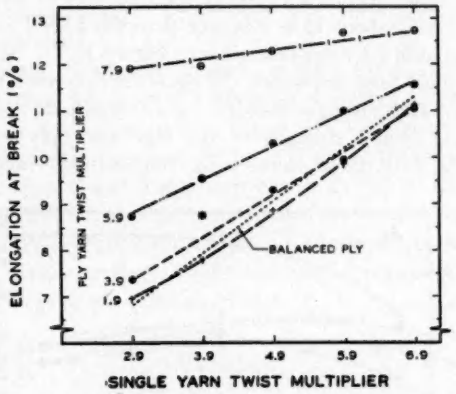

FIG. 6. *Effect of single yarn twist on elongation of a 15.5/2 carded cotton yarn.*

Equivalent Single and Ply Yarn Twist Multipliers

Figure 7 shows curves for skein and single strand strength and elongation values for equivalent single and ply twist combinations. These curves are similar to those of Figure 7 for the 31/2 yarns. In both cases maximum strength values are obtained with approximately a 4.4/4.4 twist multiplier combination for skein strength and with a 5.2/5.2 combination for single strand strength. Elongation increases as twist multiplier combinations increase, except that the rate of increase is greater for the coarser yarn.

Ply-Single Yarn Strength Ratios

To determine the gain in strength of ply over single yarns, ratios of the ply yarn count strength products to single yarn count strength products were calculated. The values plotted are based on the ply twist multiplier resulting in maximum ply yarn strength, and on the equivalent twist multiplier constructions. The largest maximum ply yarn skein strength ratio (1.48) was obtained with a 2.9 single

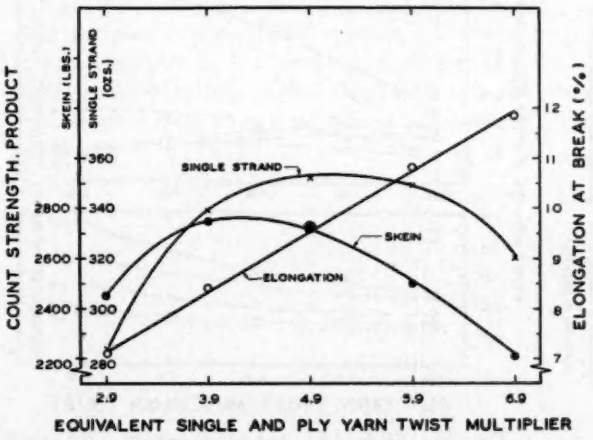

FIG. 7. *Effect of equivalent single and ply twist multiplier on skein and single strand strength and elongation of a 15.5/2 carded cotton yarn.*

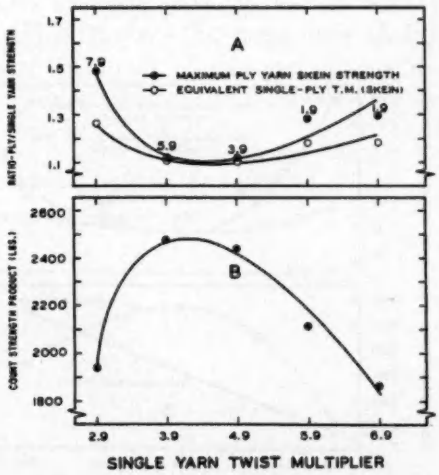

FIG. 8. A—*Skein strength ratio of 15.5/2 to 15.5/1 carded cotton yarn.* B—*Skein count strength product of a 15.5/1 yarn.*

and 7.9 ply twist multiplier construction (Figure 8A). The ratios of ply to single yarn skein strength based on equivalent single-ply twist multiplier combinations have a maximum value (1.26) with the 2.9/2.9 twist multiplier combination. These ratios (maximum strength and equivalent twist multiplier) decrease as the single yarn twist increases up to the twist multiplier (4.4 approximately) required for maximum single yarn strength (Figure 8B).

Generally, these trends are comparable to those of the 31/2 yarn, except that for low and medium twist single yarns the ratios for the 15.5/2 are smaller, indicating that the percentage gain of ply over single yarn in this area of twist becomes less as the yarn becomes coarser.

In Figure 9A the largest maximum ply yarn single strand strength ratio (1.60) was obtained with a 2.9 single and 7.9 ply twist multiplier. The 2.9/2.9 equivalent twist multiplier combination resulted in maximum ratio of 1.18. These curves are similar to those for the 31/2 yarn, with the percentage gain of ply over single yarn being less as the yarn becomes coarser.

Comparison of 15.5/1 and 31/2 Yarns

To determine the relationship between a single yarn and the equivalent 2-ply yarn, the count strength product of each single yarn (15.5/1) twist multiplier construction was divided into the count strength product of each ply twist multiplier construction (31/2) whose component single yarns (31/1) were spun with the same twist multiplier as the single yarn (15.5/1).

In Figure 10, ratios *versus* ply twist multipliers for the 31/2 yarns were plotted for a range of single yarn twist multipliers in the 15.5/1 and 31/2 yarns. In Figure 10, ratios for skein strength are shown in A, for single strand strength in B, and for elongation in C.

Using skein and single strand values as criteria, the equivalent ply yarn was generally stronger than, or equally as strong as, its single yarn counterpart for every twist construction used in this study. The skein strength of the equivalent ply yarn constructed with component single yarns spun with a 2.9 twist multiplier and then two-plied with a 2.9 twist multiplier was about 15% stronger than the 15.5/1 yarn spun with a 2.9 twist multiplier (Figure 10A). With a 6.9 ply twist multiplier, the equivalent ply yarn was about 42% stronger than its single counterpart. The single strand strength of the equivalent ply yarn made with these same twist combinations ranged

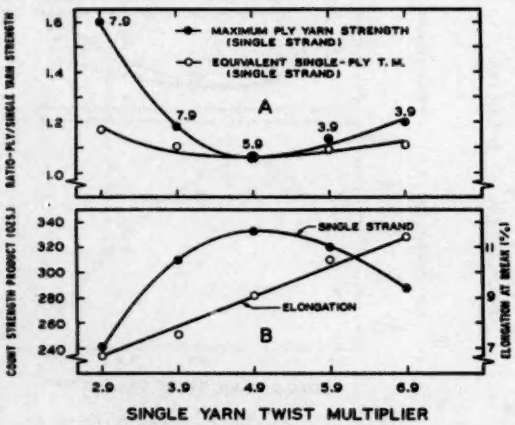

FIG. 9. A—*Single strand strength ratio of 15.5/2 to 15.5/1 carded cotton yarn.* B—*Single strand count strength product and elongation of a 15.5/1 yarn.*

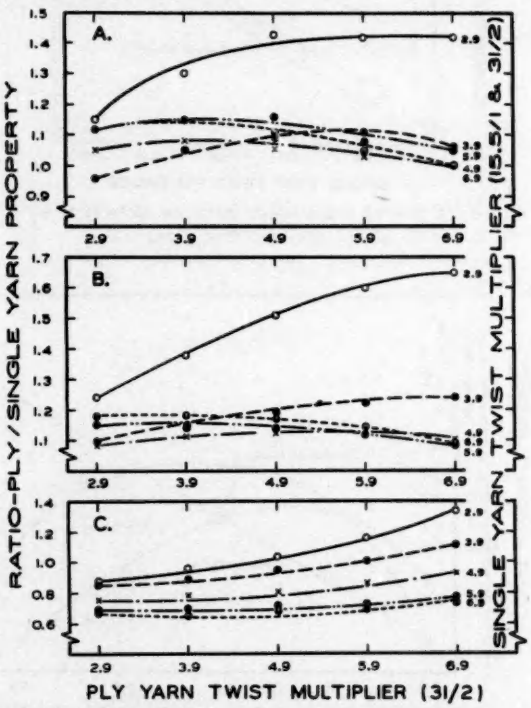

FIG. 10. *Skein* (A) *and single strand* (B) *count strength product and elongation* (C) *ratios of a single (15.5/1) yarn and an equivalent ply (31/2) carded cotton yarn.*

from 24% to 65% stronger than the single yarn (Figure 10B).

From these data, it is concluded that the strengths of equivalent ply and single yarns are dependent on the twist combinations used, and that within the limits of this study the equivalent ply yarn is generally stronger than its single yarn counterpart. It should be recognized, however, that this relationship is dependent on the processing organization, and particularly the spinning drafts used to spin the 15.5/1 and 31/1 yarns. Any organizational change which might cause a change in the strength of either single or ply yarns may invalidate the above relationships, and might in certain instances make the equivalent ply yarn weaker or stronger than its single yarn counterpart.

Using elongation values as a basis, the equivalent ply yarn was generally lower in elongation than its single yarn counterpart (Figure 10C). Exceptions to this were observed in the analysis of those yarns constructed with low twist in the single yarns (15.5/1 and 31/1) and high twist in the ply yarn (31/2). The elongation ratios of the ply to the single yarns decreased as single twist multipliers increased, regardless of ply twist multiplier.

Variability

Ply yarn variability was evaluated in terms of coefficients of variation of single strand and skein strength values. Analysis of the data gave some indication that ply yarn variability and single yarn twist are positively correlated regardless of the ply twist or method of test used—that is, ply yarns having component single yarns of high twist are more variable than those having component yarns of low twist.

Summary and Conclusions

Generally, comparisons of the properties of 15.5/2 and 31/2 yarns show similarity in strength and elongation trends for given combinations of single and ply twists. Changes from fine to coarse yarn numbers, however, cause changes in physical properties which are worthy of note.

A change to a coarse yarn causes definite differences in twist level required for maximum strength and in the characteristics of maximum strength plateaus. This change also causes areas of approximately equal yarn strengths to include a greater number of twist constructions.

Elongation values for the 15.5/2 yarns, regardless of twist constructions, are higher than for the 31/2 yarns. Also, as yarn number decreases, low single twist yarns react less sensitively elongationwise to high ply twists.

For low medium twist single yarns, the percentage gain of ply over single yarn strength becomes less as the yarn becomes coarser.

An equivalent ply yarn (31/2) is generally stronger than its single yarn counterpart (15.5/1), with strengths being dependent on the twist combinations used. On the other hand, the equivalent ply yarn is in most cases lower in elongation than its single yarn counterpart.

Ply yarn strength variability seems to increase as the twist increases in the component single yarns.

Acknowledgment

The authors wish to express sincere thanks and gratitude to fellow staff members of the Cotton Mechanical Division for processing the cotton and making the calculations and charts and to members of the Testing Section of the Analytical Division for making physical tests on the yarns.

(Manuscript received December 1, 1953.)

The Effect of Cap-Edge Friction on Spinning

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Abstract

Calculations of the effect of various spinning conditions on yarn tension and balloon size and shape were described in an earlier paper [1]. Here they are extended to include the influence of the coefficient of friction (μ) at the cap edge. Tension is found to increase as μ is increased and there is an upper limit to μ at which tension becomes theoretically infinite. There is also a lower limit to the value of μ below which balloons can only exist if they rub the side of the cap. For a given μ there is a limiting balloon height above which no free balloon is possible and this limiting height increases as μ increases. Tension can increase, decrease, or remain roughly constant as the bobbin diameter increases, depending on μ and the size of the bobbin. For a given coefficient of friction there is a bobbin size for which tension is infinite and an upper limit to the bobbin diameter above which free balloons cannot exist. The effect of friction on balloon size and shape is also indicated. The radius of the cap edge is shown to influence tension indirectly through the dependence of the coefficient of friction on the normal reaction.

Introduction

In a previous paper [1] the effect of changing various technical features in cap spinning, such as the dimensions of the spinning frame, spinning speed, yarn diameter, etc., was calculated for a perfectly smooth cap edge. The work has now been extended to show how yarn tension and balloon size depend on the coefficient of friction between the yarn and the cap edge. The calculations refer to the same idealized system as was used in the previous paper. The details are reproduced for convenience in Table I. The same assumptions regarding air drag and the equations of motion of the ballooning thread are maintained as previously, but the condition at the cap edge is modified to include frictional forces.

Condition at the Cap Edge

In [1] the following nomenclature was used: The lean-back angle θ is given by $\cos\theta = ds_1/ds$, where ds is an element of ballooning thread and ds_1 its projection on the meridian plane—*i.e.*, the plane containing the axis of the balloon and the radius, r, to the element; θ_o is the lean-back angle just above the cap edge. ϕ is the angle given by $\sin\phi = dr/ds_1$ and ϕ_o its value just above the cap edge. T_o is the tension in the balloon just above the cap edge and T_B that in the yarn between the cap edge and the bobbin, where the yarn is assumed to be straight and to lie in a horizontal plane at an angle α to the radius to the point at which the yarn leaves the cap edge. The angle α is given by $\sin\alpha = b/R$.

The velocity of the yarn along its own length is small compared with its velocity along the cap edge as it rotates in the balloon (833 cm./sec. compared with approximately 5000 cm./sec.). If we neglect the motion along the yarn and consider it simply to be moving as a whole along the cap edge, we have the problem considered by Mack [2]. He derived the following equations:

$$T_o \cos\theta_o = T_B \cos\alpha\,, \tag{1}$$

$$\tan\theta_o = \tan\alpha - \mu(\phi_o + \pi/2)\,. \tag{2}$$

The normal reaction per unit length between cap edge and yarn is N, where

$$N = (T/\rho)\cos^2\theta, \tag{3}$$

and where ρ is the radius of curvature of the cap edge, which is assumed to be the surface of revolu-

TABLE I

Height of balloon (h)	46 cm. (18.1 in.)
Radius of cap (R)	7.78 cm. (3 in.)
Radius of bobbin (b)	3.50 cm. (1.37 in.)
Spindle speed (N)	133.33 r.p.s. (8,000 r.p.m.)
Spinning speed (v)	833 cm./sec. (27.3 ft./sec.)
Speed of revolution of balloon ($\omega/2\pi$)	95.44 r.p.s. (5726 r.p.m.)
Mass of yarn per unit length (m)	6.11×10^{-5} g./cm. (55 den.)
Diameter of hypothetical yarn	0.007 cm. (0.0028 in.)

tion formed by rotating an arc of a circle of radius ρ around the axis of the balloon.

Conditions (1) and (2) can be used most simply in the following way. First, values of θ_o and ϕ_o are obtained for an assumed value of tension just below the top guide by solving the equations of motion of the ballooning thread as in the Appendix of [1] or otherwise. Then for a given bobbin size, α, the value of μ for which (2) is satisfied—*i.e.*,

$$\mu = (\tan \alpha - \tan \theta_o)/(\phi_o + \pi/2) — \qquad (4)$$

is easily found. By repeating this procedure, values of μ are obtained for a series of top tensions, and curves showing tension as a function of μ can be drawn as in Figure 1. This is more straightforward than attempting to determine top tension for a specified value of μ, a process which involves the determination by trial and error of values of θ_o and ϕ_o for which (2) is satisfied. T_o is given by the solution of the balloon equations and T_B follows from (1). Alternatively if results are required for a given value of μ and different bobbin sizes, pairs of values of θ_o and ϕ_o are again obtained for different top tensions, and for each pair the value of α for which (2) is satisfied is determined.

Calculated Results

Coefficient of Friction

Since for most balloons with which we are concerned ϕ_o is small, (2) can be written approximately as

$$\tan \theta_o = \tan \alpha - \mu\pi/2, \qquad (5)$$

which means that there is a certain value of μ—*i.e.*, $\mu = (2/\pi) \tan \alpha$—for which $\theta_o = 0$. Since this can only be obtained by having an infinitely large tension, it is a limiting value and in practice the yarn will break as the limit is approached. The limiting value is clearly greater the larger the bobbin—*i.e.*, the greater α. In Figure 1 bobbin tension is plotted against μ for different balloon heights. For a bobbin radius of 3.5 cm. and cap radius of 7.78 cm., $\tan \alpha = 0.5$ and the tension increases rapidly as μ approaches 0.3. When ϕ_o is taken into account in (4) instead of using the approximation (5), the limiting value depends slightly on balloon height. Figure 1 shows also that for balloon heights greater than approximately 70 cm. there is a minimum value of μ below which no free balloon exists. The reason for this is essentially the same as was advanced in the earlier paper [1] to account for the limiting height above which no free balloon is possible but only one rubbing the side of the cap. Thus equation (2) requires θ_o to increase as μ decreases, other factors remaining unchanged. But it was shown in [1] that there is a limiting lean-back angle which cannot be exceeded for given spinning conditions, whatever the tension. This means that if μ is less than a certain value, the lean-back angle required for equilibrium at the cap edge is greater than the forces acting on the ballooning thread are able to produce, and hence no stable, free balloon can be set up. As the balloon height decreases so does the lower limit to μ. For heights less than 70 cm. it is theoretically negative, so that in practice no lower limit to μ exists for small heights.

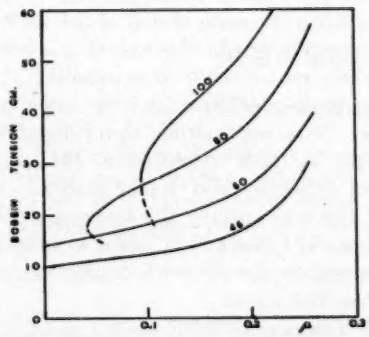

FIG. 1. *Effect of friction on bobbin tension. The numbers on the curves are balloon heights in cm.*

Balloon Height

In [1] it was found that for a perfectly smooth cap edge there is a limiting height greater than which no free balloon can exist. Figure 2 shows that a limiting height exists also for a rough cap and that

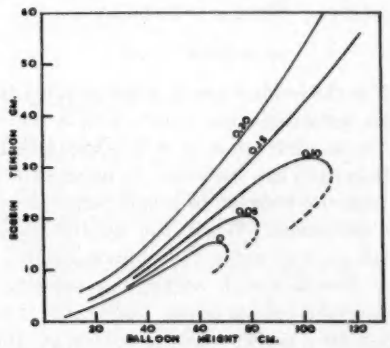

FIG. 2. *Effect of balloon height on bobbin tension. The numbers on the curves are values of the coefficient of friction.*

the rougher the cap edge the greater the limiting height. The limit may of course be outside the range of normal operating conditions if μ is large enough.

Bobbin Diameter

Figure 3 shows for different values of μ at the cap edge how bobbin tension varies as the bobbin diameter changes. It appears that tension can increase, decrease, or remain constant as the bobbin diameter increases, according to the value of μ and the bobbin size. Thus taking the curve for $\mu = 0.25$ as an example, tension is very large when the bobbin diameter is about 6 cm. and decreases as the bobbin gets bigger. An infinite tension is to be expected from (2) when $\tan \alpha = \mu (\phi_o + \pi/2)$, since for this value of α, $\theta_o = 0$. Remembering that $\sin \alpha = b/R$ and that ϕ_o is small compared with $\pi/2$, it follows that the bobbin diameter at which the tension becomes very large is smaller the smoother the cap edge. This is clearly illustrated in Figure 3. For all values of μ there is a limiting bobbin size above which no balloon can exist which does not rub the side of the cap, the limiting size increasing as the cap edge gets rougher. This also can be explained by the inability of the forces on the balloon to produce a lean-back angle large enough to give equilibrium at the cap edge, the necessary lean-back increasing as bobbin size increases.

Curves of Figure 3 for low values of μ—*e.g.*, $\mu = 0$ and 0.05—show a different behavior from that for $\mu = 0.25$. Tension is again very large for a certain size of bobbin, but falls to zero when the bobbin diameter is 2 cm. This is the diameter for which the angular velocity ω of the ballooning thread is zero. Since ω is given by

$$\omega = 2\pi N - v/b, \qquad (6)$$

where N is the bobbin speed, v the velocity at which the yarn leaves the top godet, and b the bobbin radius, it is clear that $\omega = 0$ when $b = v/2\pi N$. For bobbin radii less than this, an increase in bobbin size causes the balloon to rotate more slowly and tension decreases. When the bobbin radius exceeds this critical value the balloon rotates in the opposite direction with an angular velocity which increases as the bobbin grows, leading to an increase in tension for a while. As the bobbin continues to get larger the yarn between the cap edge and the bobbin becomes more and more inclined to the radius, an effect which tends to reduce tension.

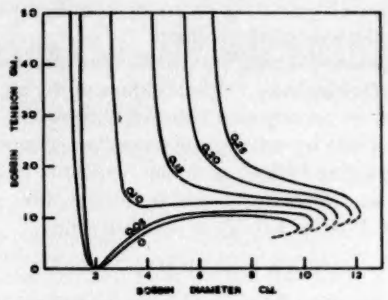

FIG. 3. *Effect of bobbin diameter on bobbin tension. The numbers on the curves are values of the coefficient of friction.*

The behavior indicated in Figure 3 for very small bobbins may well be of academic interest only but it is included here for completeness.

Other Spinning Variables

We have seen that there is a limiting height above which no free balloon is possible and a corresponding limiting bobbin size. These we have attributed to the fact that there is an upper limit to the lean-back angle that the forces on the ballooning yarn can produce. Inspection of all the calculated results obtained reveals that the maximum lean-back angle occurs when $T_z/mh^2\omega^2 = 0.12$ approximately (T_z is the vertical component of tension in the balloon and nomenclature is otherwise as in Table I), which is slightly less than the value for which the balloon develops a concavity above the cap edge and for which the maximum balloon diameter occurs [1]. Results for different deniers reveal that there is also a certain high denier above which no free balloon is possible. Similarly there is a limiting value of cap diameter when the ratio of cap to bobbin diameter is kept constant, but in this case it is a lower limit below which no free balloon is possible. It is not proposed to discuss these limiting cases in detail, since they lie outside normal operating conditions, but simply to draw attention to their existence. The effect on tension of a change in denier, etc., for a rough cap is essentially the same as shown for a smooth one in Figures 1, 3, and 4 of reference [1] with the tension everywhere increased according to Figure 1 of this paper.

Figure 4 shows the influence of cap-edge friction on the balloon profile. A concavity develops when μ is near the lower limiting value for which a free

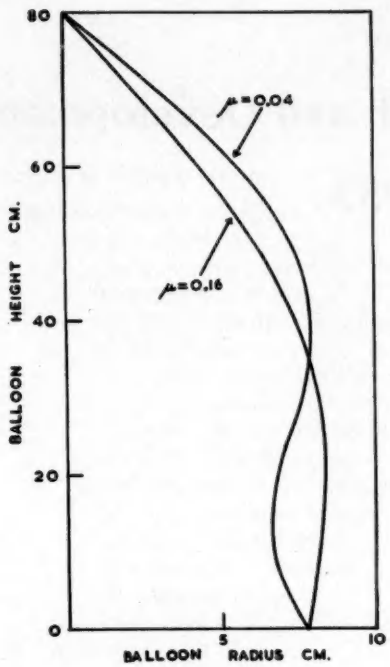

FIG. 4. *Balloon profiles for two values of the coefficient of friction.*

balloon is possible. The diameter of the balloon is not very sensitive to the value of μ.

So far it has been implicitly assumed that the coefficient of friction is constant for a given yarn and cap. In many cases this is possibly a good approximation but in others it appears that μ depends on the normal reaction and the speed with which the yarn moves over the surface. There is some published evidence, for example, that μ decreases as N increases. If this is the case, when a change in tension is produced by altering the balloon height, for example, the normal reaction, N, also changes according to equation (3) and hence the coefficient of friction assumes a different value. Thus the change in μ which accompanies any alteration in spinning conditions needs to be allowed for when estimating the effect of changing any technical feature. The results calculated in this paper must thus be regarded as first approximations to the behavior to be expected in practice.

The dependence of μ on the normal reaction is particularly important when the effect of the radius of the cap edge is considered. If μ is constant the cap-edge radius ρ affects only the normal component N through (3) and has no influence on tension or balloon size. If μ depends on N, however, then an alteration in ρ changes N and hence μ. Thus if μ decreases with increasing N, a smaller cap-edge radius is equivalent to a smaller coefficient of friction and produces a reduced tension as in Figure 1. According to the present theoretical treatment it is through this indirect effect on the coefficient of friction that the cap-edge radius influences yarn tensions and balloon properties.

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(*Manuscript received October 15, 1953.*)

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Review of Textile Research and Development During 1953

Edited by

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Textile Research Institute, Princeton, New Jersey

Table of Contents

Page

Introduction . . . 439

Bibliography . . . 439

I. **Papers of General Interest** . . . 439

II. **Cellulose Fibers** . . . 439

A. *Chemistry of Cellulose* . . . 439

1. Chemical Structure of Cellulose . . . 439

2. Reactions of Cellulose . . . 440

(*a*) Oxidation and Decomposition . . . 440

(*b*) Hydrolysis . . . 440

(*c*) Preparation and Properties of Cellulose Ethers . . . 441

(*1*) Carboxymethyl Cellulose . . . 441

(*2*) Methyl and Ethyl Cellulose . . . 441

(*3*) Miscellaneous . . . 441

(*d*) Preparation and Properties of Cellulose Esters . . . 441

(*1*) Cellulose Acetate . . . 441

(*2*) Miscellaneous . . . 442

(*e*) Reaction with Alkali . . . 442

(*f*) Miscellaneous . . . 443

3. Analysis of Cellulose and Cellulose Derivatives . . . 443

B. *Fine Structure Cellulose* . . . 443

C. *Mechanical Properties of Cellulose Fibers* . . . 444

D. *Absorption and Swelling of Cellulose* . . . 444

E. *Cotton Fibers* . . . 445

F. *Bast and Hard Fibers* . . . 445

1. Jute . . . 445

2. Other Bast Fibers . . . 446

3. Hard Fibers . . . 446

G. *Rayon Fibers* . . . 446

1. Viscose Process . . . 446

(*a*) Pulp Characterization and Reactions with Alkali . . . 446

(*b*) Xanthation . . . 446

(*c*) Properties of Viscose Solutions . . . 447

(*d*) Regeneration . . . 447

2. Acetate Process . . . 447

3. Structure . . . 448

III. **Protein Fibers** . . . 449

A. *Wool and Other Hair Fibers* . . . 449

1. General . . . 449

2. Morphology . . . 449

3. Fine Structure . . . 449

4. Chemical Reactions . . . 450

5. Mechanical Properties . . . 451

6. Absorption and Swelling . . . 451

7. Damage . . . 452

8. Wool Wax . . . 452

B. *Silk* . . . 452

C. *Regenerated Protein Fibers* . . . 453

Page

IV. Synthetic Fibers ... 453
A. General ... 453
B. Polycondensation Polymers ... 453
1. Polyamide Fibers ... 453
(*a*) Polyhexamethyleneadipamide ... 453
(*b*) Poly-ε-aminocaprolactam ... 454
(*c*) Miscellaneous ... 455
2. Polyester Fibers ... 455
C. Addition Polymers ... 455
1. Polyvinyl Fibers ... 455
(*a*) Polyvinylchloride ... 455
(*b*) Polyacrylonitrile ... 455
(*c*) Polyvinylalcohol ... 456
2. Mixed Polymers ... 457
D. Miscellaneous ... 457

V. High-Polymer Studies ... 458
A. Mechanical Properties and Structure ... 458
B. Properties of High-Polymer Solutions ... 458
C. Absorption, Swelling, and Diffusion ... 459

VI. Molecular-Weight Determination ... 460
A. Viscosity ... 460
1. General and Theoretical Papers ... 460
2. Methods and Instruments ... 460
B. Osmotic Pressure ... 461
C. Sedimentation and Diffusion ... 461
D. Light Scattering ... 461
E. Fractionation ... 462

VII. Deterioration of Materials ... 462
A. Microbiological Degradation ... 462
B. Effects of Light ... 463
C. Moth Damage and Mothproofing ... 463

VIII. Detergency and Surface-Active Agents ... 463
A. Detergency ... 463
1. General ... 463
2. Measurement ... 464
3. Builders ... 464
4. Micelle Formation ... 464
5. Adsorption ... 465
B. Behavior of Fabrics in Laundering and Dry Cleaning ... 465
C. Wetting ... 465
D. Analysis of Surface-Active Agents ... 465

IX. Test Methods and Apparatus ... 466
A. Geometrical and Mechanical Properties ... 466
1. Single Fibers ... 466
(*a*) Length and Diameter ... 466
(*b*) Mechanical Properties ... 466
(*c*) Optical and Other Properties ... 467

Page

2. Fiber Assemblies ... 467
(a) Mechanical Properties of Yarn ... 467
(b) Mechanical Properties of Fabric ... 467
(c) Other Fiber Assemblies ... 468
(d) Numbering Systems ... 468
B. *Chemical Tests*
1. Identification of Fibers ... 468
2. Analysis of Fibrous Materials ... 468
3. Determination of Moisture ... 469
C. *Studies with Radiation* ... 469
1. Microscopy and Photography ... 469
2. Spectrophotometry and Color ... 469
3. Nuclear Radiation and X-Ray ... 469
D. *Statistical Topics* ... 469
E. *Miscellaneous* ... 470

X. **Fiber-Yarn-Fabric Relationships** ... 470
A. *Fiber Properties and Their Relation to Properties of Fiber Assemblies* ... 470
B. *Interfiber Friction* ... 470
C. *Regularity of Sliver, Roving, and Yarn* ... 471
D. *Effect of Twist on Yarn* ... 471
E. *Fabric Behavior* ... 471

XI. **Manufacturing** ... 472
A. *General* ... 472
1. Raw Materials ... 472
(a) Protein Fibers ... 472
(b) Cellulose Fibers ... 472
(c) Synthetic Fibers ... 472
2. Operation of Mills and Machinery ... 473
3. Processing of Blends ... 473
4. Static Electricity ... 473
B. *Preliminary Processes* ... 474
1. Opening and Blending ... 474
2. Wool Scouring ... 474
C. *Carding* ... 475
D. *Drafting and Spinning* ... 475
1. Drafting ... 475
2. Spinning ... 475
3. Winding and Twisting ... 476
E. *Weaving* ... 477
1. Warp Sizing ... 477
2. Advances in Loom Construction ... 477
3. Studies of Loom Operation ... 477
F. *Knitting* ... 478
G. *Nonwoven Fabrics* ... 478

XII. **Dyeing and Finishing** ... 478
A. *General* ... 478
B. *Preliminary Processes* ... 479
1. Desizing ... 479
2. Bleaching and Stripping ... 479
3. Miscellaneous ... 480

Page

C. Dyeing
1. New Dyes ... 480
2. Theoretical Papers ... 480
(*a*) Properties of Dyes ... 480
(*b*) Interaction of Dyes and Fibers ... 481
3. Protein Fibers ... 482
4. Cellulosic Fibers ... 482
(*a*) Cotton and Viscose Rayon ... 482
(*b*) Acetate Rayon ... 483
5. Synthetics ... 483
6. Blends ... 484
7. High-Temperature Dyeing and Other New Developments ... 485
(*a*) High-Temperature Processes ... 485
(*b*) New Methods in Vat Dyeing ... 485
(*c*) Other New Processes ... 485
8. Testing ... 485
(*a*) Color Measurement and Matching ... 485
(*b*) Fastness Determination ... 486
D. Printing ... 486
E. Finishing ... 487
1. Milling ... 487
2. Crease-Resistant Finishing ... 487
(*a*) Research ... 487
(*b*) Development ... 488
(*c*) Analysis and Testing of Treated Fabrics ... 488
3. Flameproofing ... 489
4. Fabric Setting ... 489
5. Water-Repellent Finishing ... 489
6. Miscellaneous ... 489
F. Drying ... 490
G. Water Supply and Waste Disposal ... 490

ANNUAL REVIEW OF TEXTILE RESEARCH AND DEVELOPMENT FOR 1954

The 1953 Review appearing in this issue will be the last Review to be published in *Textile Research Journal*. The 1954 Review will be available in a separate cover in the spring of 1955 at a prepublication price of $5.00 per copy. Orders for the 1954 Review should be addressed to Mr. P. C. Alford, Jr., Secretary, Textile Research Institute, Princeton, New Jersey, and should be accompanied by check or money order.

This change is occasioned by the conclusion of support of this Review by the Office of Naval Research.

Review of Textile Research and Development During 1953

Introduction

This is the fifth annual review of the literature in the field of textile research and development prepared and published by the Textile Research Institute. Continuous effort to make the *Review* more useful and more complete has resulted in the present form of the work—a bibliography divided into subject classifications with cross-references. The main subject divisions are essentially the same as those in the *Review* published in 1953.

The references selected by the T.R.I. staff members have been carefully reviewed by the editors—in most cases by examination of the original articles—to confirm the relevance of the papers to the subjects being covered and to classify and cross-reference them correctly. Many articles, particularly anonymous ones, were rejected at this stage.

Great care has been taken to insure the accuracy of the bibliography. Where journals have not been available for checking, the abstract references have been given in parentheses after the original citations. Abstract references have also been included for all items from the Russian literature and many from the Japanese. As in the past four *Reviews*, a few textile subjects have systematically been omitted, and no patent literature has been included. These omissions were necessary in order to keep the *Review* to a manageable size.

The task of searching the literature and making the initial selection of papers was carried out by the following members of the staff of Textile Research Institute: J. H. Dusenbury, C. Messler, D. J. Montgomery, R. F. Schwenker, Jr., R. Steele,* R. K. Toner, H. Wakeham, J. H. Wakelin, and H. J. White, Jr. The important and arduous task of preparing the bibliographic cards from which the manuscript was prepared has again been very ably done by Mrs. Charles Rosenblum.

The Institute wishes to express its appreciation to the Office of Naval Research for support of this *Review* and for permission to publish it.

* Dr. Steele was able to assist with the *Review* through the cooperation of the Rohm and Haas Company.

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V. HIGH-POLYMER STUDIES

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VI. MOLECULAR-WEIGHT DETERMINATION

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VI. MOLECULAR-WEIGHT DETERMINATION

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VII. DETERIORATION OF MATERIALS

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VIII. DETERGENCY AND SURFACE-ACTIVE AGENTS

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VIII. DETERGENCY AND SURFACE-ACTIVE AGENTS

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IX. TEST METHODS AND APPARATUS

A. Geometrical and Mechanical Properties

1. Single Fibers

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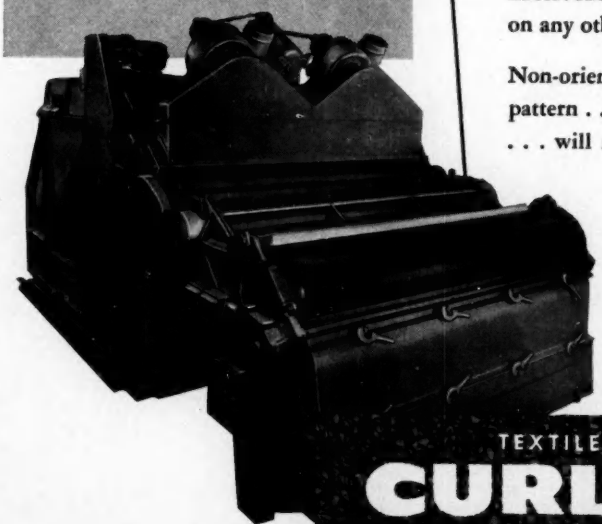

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